ERDC/CRREL TR-10-1

Cold Regions Research
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Adsorption/Desorption Measurements of Nitroglycerin and Dinitrotoluene in Camp Edwards, Massachusetts Soil

Jay L. Clausen, C. Scott, N. Mulherin, S. Bigl, G. Gooch,
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February 2010

Cover photo: Experimental setup for the adsorption/desorption column experiments.

U.S. Army Corps of Engineers
New England District

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Abstract: The mobility potential of nitroglycerin (NG) and dinitrotoluene (DNT) in small-arms range (SAR) soils was studied through a determination of adsorption and desorption. Measured laboratory batch-adsorption soil/water partitioning coefficients (K_ds) for 2,4-DNT, 2,6-DNT, and NG ranged from 0.1 to 21.3, 0 to 18.2, and 0 to 7.3 L/kg, respectively. Mean adsorption K_d for 2,4-DNT and 2,6-DNT were 3.2 and 2.6 L/kg, respectively. The mean value for NG was 0.9 L/kg. The variables impacting adsorption were organic matter and cation exchange.

Unfired and fired propellant tests suggest that NG and DNT is not completely available for dissolution, and tests with weathered soils indicate none of the NG is available, although analysis shows NG is still present in the soil. Dissolution is the most important process in describing migration of deposited propellant compounds from SARs. Once released from nitrocellulose and dissolved in water, adsorption and degradation processes further limit NG and DNT mobility.

Column experiments were conducted to augment batch tests. Nearly instantaneous breakthrough of NG was evident for the pair of columns containing aqueous NG/DNT with biocide. Results for the columns containing aqueous NG/DNT without biocide, fresh-fired propellant residue with biocide, and fresh-fired propellant residue without biocide indicated no breakthrough.

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Nomenclature

1,2-GDN	glycerol-1,2-dinitrate
1,3-GDN	glycerol-1,3-dinitrate
1-GMN	glycerol-1-mononitrate
2-GMN	glycerol-2-mononitrate
ASTM	American Society for Testing and Materials
MDL	method detection limit
bgs	below ground surface
CEC	cation exchange capacity
CENAE	U.S. Army Corps of Engineers, New England District
CFB	Canadian Force Base
CRREL	U.S. Army Corps of Engineers, Cold Regions Research and Engineering Laboratory
DI	deionized water
DNT	dinitrotoluene
ERDC	U.S. Army Corps of Engineers, Engineer Research and Development Center
GC-ECD	gas chromatography – electron capture detector
GDN	dinitroglycerin
GMN	mononitroglycerin

HMX	octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
HPLC	high performance liquid chromatography
ICP-OES	Inductively Coupled Mass Spectrometry
K_d	soil/water partitioning coefficient
K_{oc}	organic carbon adsorption coefficient
log K_{ow}	log octanol water partitioning coefficient
MMR	Massachusetts Military Reservation
MDL	method detection limit
NC	nitrocellulose
NG	nitroglycerin
OC	organic carbon
RDX	hexahydro-1,3,5-trinitro-1,3,5-triazine
RPD	relative percent difference
SAR	small arms range
SERDP	Strategic Environmental Research Program
SPE	solid phase extraction
TNT	2,4,6-trinitrotoluene
UASCE	U.S. Army Corps of Engineers
USEPA	U. S. Environmental Protection Agency

Preface

This report was prepared by Jay L. Clausen, Biogeochemical Sciences Branch (BSB), Engineer Research and Development Center (ERDC), Cold Regions Research and Engineering Laboratory (CRREL), Hanover, New Hampshire, and Dr. Ian T. Osgerby, U.S. Army Corps of Engineers (USACE), New England District, Geo-Environmental Engineering Branch, Geology/Chemical Section.

The U S. Army Environmental Command Impact Area Groundwater Study Program (IAGWSP) provided funding for this work through USACE, New England District. Dr. Charles M. Reynolds and Dr. David Ringleberg, CRREL, provided technical reviews.

This report was prepared under the general supervision of Terrence Sobecki, Branch Chief, BSB, CRREL; Dr. Justin Berman, Chief, Research and Engineering Division, CRREL; and Dr. Robert E. Davis, Director, CRREL. The Commander and Director of the ERDC is Col. Gary E. Johnston. The Director is Dr. Jeff Holland.

Acknowledgement is given to Mike Kulbersh and Mark Koenig for assistance in Quality Assurance/Quality Control checks of the data, to Robert Cannata and Megan Cullen for data entry assistance, USACE, New England District, and to the several reviewers who represented the IAGSWP, the EPA, and MADEP for helpful and constructive comments.

Unit Conversion Factors

Multiply	By	To Obtain
cubic feet	0.02831685	cubic meters
cubic inches	1.6387064 E-05	cubic meters
degrees Fahrenheit	(°F-32)/1.8	degrees Celsius (°C)
feet	0.3048	meters
gallons (U.S. liquid)	3.785412	Liters
liters	1.0 E-3	cubic meters
inches	0.0254	meters
pounds (mass)	0.45359237	kilograms
pounds (mass) per cubic foot	16.01846	kilograms per cubic meter

Executive Summary

This laboratory study evaluated the mobility potential of nitroglycerin (NG or trinitroglycerine) and dinitrotoluene (DNT) in small-arms range (SAR) soils at Camp Edwards. The objectives were to determine the adsorption and desorption as NG and DNT interact with soil. The information derived is useful in providing guidance when developing loading rates and applicability of soil partitioning coefficients (K_d) for predicting transport of these compounds from surface soil through the unsaturated zone. The results of evaluation of dissolution of these compounds by Taylor (CRREL) are attached as Appendix A. These results indicate that dissolution is the most important process in describing migration of deposited propellant compounds from training exercises at SARs.

Double-base smokeless powders typically contain nitrocellulose (NC), NG, stabilizing agents, and filler compounds. During the manufacturing process, dinitroglycerin and mononitroglycerin are produced as impurities, and DNT is often added as a flash suppressor. Reagent-grade NG is somewhat soluble in water, and transport models such as SESOIL suggest rapid movement of NG. NG and DNT are encapsulated in the NC during manufacture of the propellants. However, NC is not soluble in water, and the physical release of NG and DNTs from the NC matrix after firing relies upon the physical weathering of NC and subsequent dissolution. Once released from the NC and dissolved in water, adsorption and degradation processes further limit NG and DNT mobility.

Adsorption and desorption batch experiments were conducted with aqueous, reagent-grade NG and DNT in deionized water (DI), fresh-unfired and fired propellant, and soil contaminated with weathered-fired propellant. Assessed experimental conditions included effects of inter-and intrasite heterogeneity, depth, soil pH, temperature, precipitation versus DI, and reagent-grade contaminant concentration. Adsorption experiments were initially conducted using a range of NG concentrations (0.1, 1, 10, 40, and 80 mg/L) to evaluate linearity of the adsorption isotherm process. Linearity was demonstrated, and all subsequent batch adsorption experiments were conducted with 10 mg/L reagent-grade NG to enhance analytical accuracy.

A series of tests were carried out to determine the duration of the batch adsorption tests required to reach steady-state conditions. Durations were evaluated from 24 hr to greater than 216 hr with 24 hr selected as sufficient for most observations. All adsorption batch tests were then conducted at room temperature for 24 hr using 10 mg/L reagent-grade NG/DNT with biocide. The measured laboratory batch adsorption K_d values for 2,4-DNT, 2,6-DNT, and NG ranged from 0.1 to 21.3, 0 to 18.2, and 0 to 7.3 L/kg, respectively. The mean adsorption K_d for 2,4-DNT is estimated to be 3.2 L/kg and 2.6 L/kg for 2,6-DNT. The mean value for NG is estimated to be 0.9 L/kg. The only variable that appeared to have a significant impact on adsorption values was soil depth, which may be related to organic matter or cation exchange capacity, or a combination of the two.

All desorption batch tests were also conducted at room temperature for 24 hr with DI. The desorption K_d values for 2,4-DNT, 2,6-DNT, and NG ranged from 0 to 11.6, 0 to 33.1, and 0 to 10.1 L/kg, respectively. The mean desorption K_d for 2,4-DNT and 2,6-DNT is estimated to be 4.9 L/kg and 5.7 L/kg, respectively. The mean desorption K_d for NG is estimated to be 1.6 L/kg. Similar to the adsorption results, only soil depth appeared to have a bearing on the desorption values. The NG desorption results suggest that although a steady state was generally demonstrated, equilibrium for some samples may not have been fully achieved within the 24-hr test period, resulting in a potential overestimation of desorption K_d values.

Desorption was not measurable from contaminated soil containing weathered fired propellant as neither DNT nor NG were detected in the aqueous phase. NG was detected in the aqueous phase in tests with freshly fired propellant and unfired propellant with uncontaminated soil, but only with the addition of a biocide. In contrast, the same experiments with fresh-fired propellant without application of a biocide resulted in undetectable NG in the aqueous phase.

A batch test was also conducted with an aliquot of fresh-fired and unfired propellant but with no soil present in the mix. This test was conducted to evaluate the portion of the total NG in the propellant mass readily soluble in water, and hence, available for adsorption/desorption/biodegradation. Only a limited portion of the total mass of NG associated with the freshly-fired and unfired propellant dissolved in water over 24-hr. This result is similar to the results of the dissolution study conducted by Taylor (CRREL) (Appendix A).

Batch studies consistently indicated that desorption K_ds were higher with overall mean values higher by a factor of two to three when compared to the adsorption K_d values. Individual values varied significantly (as already noted), but the magnitude of the variation may be within the margin of experimental error. Additionally, results of batch tests with and without biocide indicate biodegradation could significantly limit or potentially eliminate migration through the unsaturated zone.

Column experiments were conducted to augment batch adsorption/desorption tests and consisted of eight identical columns assessing four scenarios: 1) aqueous NG/DNT with biocide; 2) aqueous NG/DNT without biocide; 3) fresh-fired propellant residue with biocide; and 4) fresh-fired propellant residue without biocide. Each scenario was assessed in duplicate using paired columns. All column experiments were loaded with 20-cm soil heights. For Scenarios 3 and 4, fresh-fired propellant was placed on top of the soil column at an estimated loading of 1,120 51 mg/kg NG (11.20%) of the total mass of 0.14 g of fired propellant. The actual loading of available NG was not determined from the total mass of NG in the NC.

In Scenario 1, nearly instantaneous breakthrough of NG was evident for the pair of columns with aqueous NG/DNT with biocide. Near-peak NG concentration was achieved in both columns by 24 hr. The peak concentration was 85 to 90% of the total NG concentration introduced to the columns. The breakthrough of DNT lagged several hours from initial startup. The DNT effluent concentration increased rapidly within the first 50 hr, followed by a slower increase. A mass balance was not achieved even after 960 hr.

Results for the columns containing aqueous NG/DNT without biocide (Scenario 2), fresh-fired propellant residue with biocide (Scenario 3), and fresh-fired propellant residue without biocide (Scenario 4) indicated no breakthrough of NG and DNT. The column results for aqueous NG/DNT without biocide suggest biodegradation processes may be consuming NG and DNT. Biodegradation daughter products were not detected, suggesting that the degradation process does not stop at intermediate products. The lack of NG or DNT detected in the effluent for the column experiments in Scenario 3 suggest dissolution is likely the rate-limiting step for contaminant release on SARs. The results reported here show adsorption/desorption/biodegradation processes are more than adequate to

handle the limited NG and DNT released into the aqueous phase by dissolution (Appendix A).

The K_d values derived with the aqueous, reagent-grade NG and DNT are appropriate for modeling purposes for the portion of DNT and NG dissolved in the environment during precipitation events. Experimental results clearly indicate that partitioning is reversible, which is a key assumption with typical (SESOIL) unsaturated zone models. However, results with unfired and fired propellant suggest that not all of the NG and DNT present in the propellant are available for dissolution and subsequent interaction with the soil. Results with weathered soils indicate none of the NG residual is available for dissolution, although analysis shows NG is still present in the soil. This finding is consistent with the results of tests carried out by Speitel et al. (2002) at the University of Texas. Therefore, modeling should not strictly focus on the adsorption/desorption partitioning values but should also consider the effects of dissolution and biodegradation. Separate, ongoing tests of dissolution of fired and unfired propellants (Appendix A) indicate only ~ 5% of the total NG in the NC matrix in fired propellant is actually available (~ 2.5% of the total in unfired propellant). This limited dissolution occurs in the first few months of exposure to weathering, followed by dissolution, and is apparently limited to diffusion from the bulk interior to exposed surfaces.

1 Introduction

Double-base propellants used within newer small-arms ammunition typically contain up to 84% nitrocellulose (NC), with 10% nitroglycerin (NG), a stabilizer, and up to 6% filler compounds. Dinitroglycerin (GDN) and mononitroglycerin (GMN) are impurities produced in the manufacture of NG, and dinitrotoluene (DNT) is often added as a flash suppressor.

NG and DNT have been detected in surface soils at small-arms range (SAR) firing points, and DNTs have been identified at gun and mortar firing points at Camp Edwards. Although there is some evidence of DNTs leaching to groundwater at Demolition Area 1, there is no current evidence of NG and its degradation (daughter) products leaching from soil to groundwater at Camp Edwards SARs. However, a concern remains that these compounds may migrate to groundwater in the future.

Numerous processes affect the fate-and-transport of NG and DNT from surface soil through the unsaturated zone. The dissolution of NG and DNTs from NC and surface soil is not well understood and is currently being researched at the Engineer Research Development Center- (ERDC) Cold Regions Research and Engineering Laboratory (CRREL) with ongoing drip tests (Lever et al. 2005; Dontsova et al. 2006; see Appendix A for current NG and DNT research). Commercial models are available to simulate this process but lack specifics of dissolution process mechanics and degradation mechanisms. Therefore, evaluations of the fate and transport of compounds through the unsaturated zone often overlook the dissolution and degradation steps and focus on the partitioning of the compounds at the soil/water interface. A key variable in such an analysis is the partitioning coefficient (K_d) for the compound of concern. K_d determines the maximum quantity of contaminant adsorbed to (or desorbed from) a specified mass of contaminated soil and volume of liquid and is defined as follows:

$$K_d = \frac{S}{C_e} = \frac{C_0 - C_e}{C_e} \times \frac{V}{m} \quad 1\}$$

where:

K_d = soil-to-water partition coefficient for a given substance (L/kg)
S = mass of sorbed contaminant per mass of soil (mg/kg)
C_e = equilibrium liquid phase concentration (mg/L)
C_0 = initial liquid phase concentration (mg/L)
V = volume of the liquid phase (L)
m = mass of soil (kg)

1.1 Nitroglycerine

Reagent-grade NG is soluble in water at approximately 1,250 to 1,950 mg/L (Rosenblatt et al. 1991; Windholz 1976). Consequently, when transport models such as SESOIL are used without considering factors other than solubility, the calculated results suggest rapid movement of NG through the soil horizon. However, NG is susceptible to adsorption, rapid photo degradation, and biodegradation.

Early work by Urbanski (1964) suggested NG photo degradation was possible and later work conducted by Rosseel et al. (1974) suggested the rate of loss via this mechanism was insignificant. Subsequent work by Spanggord et al. (1980) suggested a slow rate of photolysis yielding a half life of 57 to 73 days. Using the U.S. Environmental Protection Agency (USEPA) subprogram in Atmospheric Oxidation Windows Program in EPI Suite (USEPA 2007) software yields a photo stability estimate of 117 hr or approximately 10 days with 12 hr of daylight per day.

Aerobic biodegradation studies utilizing static batch tests by Jenkins et al. (2003) found the half - life of NG to be less than 1 day; which was so rapid that the loss rate could not be quantified. Stirred batch reactor studies by Yost (2004) with a clay-loam soil and organic carbon content of 0.2%, yielded a NG half- life of 1.5 days. Similarly, studies by Lyman et al. (1982) found NG to have a half - life of 2 to 7 days. Nitroglycerin degradation follows successive denitration to produce glycerol 1,2- and 1,3-dinitrate (1,2-GDN and 1,3-GDN), and glycerol 1- and 2-mononitrate (1-GMN and 2-GMN). The GDN and GMN isomers further breakdown into glycerol and carbon dioxide (Dacre and Rosenblatt 1974). However, these metabolites were not found in a study conducted by Spanggord et al. (1980) who speculated that the rate of transformation of these metabolites was so rapid that they do not accumulate. GDN isomers have explosive properties similar to the NG parent compound (Urbanski 1965). Spanggord et al. (1980) showed 96% conversion of NG to nitrite, and microorganisms can utilize NG as a sole carbon source. Studies by Marshal and White (2001),

Bhaumik et al. (1997), and Blehert et al. (1997) have identified a variety of bacterium NG degraders. Spanggord et al. (1980) showed 96% conversion of NG to nitrite. Untransformed NG is readily susceptible to soil sorption processes.

Previous batch adsorption experiments with reagent-grade NG in Camp Edwards soils suggested an average K_d value of approximately 2 L/kg (Speitel et al. 2002). In contrast, no desorption was observed to occur in batch desorption tests with weathered contaminated soils (Speitel et al. 2002). Although nothing was detected in the final solution, a desorption $K_d > 71$ L/kg was estimated based on an initial soil concentration of 7,120 µg/kg, assuming it was present at the detection limit (100 µg/L). Speitel et al (2002) and Yamato et al (2004) are the only published studies to have evaluated the K_d of NG.

Rapid attenuation of NG from propellant in soil is consistent with in-progress soil studies at SARs at Yakima Training Range, Fort Lewis, Fort Richardson, and CFB-Petawawa (Jenkins et al. 2007; Hewitt 2007; Brochu et al. 2006). These studies indicate NG residues are deposited on the surface soil at SAR firing points, with concentrations up to 504 mg/kg. At Fort Richardson, a typical soil profile yields the highest NG concentrations in the 0 - to 2 - cm interval, with declining concentrations with increasing depth. In most borings, NG attenuation occurred by 15 cm (Hewitt 2007).

In addition to studies at SAR firing points, more extensive research has focused on the deposition of NG residue at rocket, mortar, and artillery firing points (Jenkins et al. 2007, 2004, 2001; Brochu et al. 2006; Thiboutot et al. 2004, 2003; Pennington et al. 2003, 2002). Nitroglycerin has been found in surface soil samples at artillery/mortar firing points but is not generally seen in deeper soil samples. Lysimeters installed 2 ft below ground surface at an artillery firing point had no detectable NG despite the presence of NG in the surface soil (Jenkins et al. 2007). Studies conducted at anti-tank rocket firing points at Yakima Training Center, Washington (Pennington et al. 2002); Fort Bliss, Texas (Pennington et al. 2003); Canadian Force Base (CFB) Gagetown (Thiboutot et al. 2003, 2004); CFB Valcartier (Jenkins et al. 2004); and CFB Petawawa (Brochu et al. 2006) indicate NG is present with the highest concentrations and percent levels at locations behind the firing line, due to the back-blast of shoulder-fired rockets. NG residues were concentrated in the surface soil. At CFB Petawawa, an extensive array of monitoring wells has been installed at an anti-

tank rocket range to track the presence of octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) in groundwater. Although, NG is present in surface soils it has not been observed in groundwater, which is consistent with NG being observed in soil at a maximum depth of 60 cm (Thiboutot et al. 2004).

These findings are consistent with numerous studies conducted at Camp Edwards. Of 12,376 soil samples analyzed for NG, there were 166 positive detections. None of the NG detections were present in soil samples deeper than 8 ft, and 161 of the NG detections were found in the top 2 ft of the surface soil. At the KD Rocket Range, NG was seen in surface soils up to 130 mg/kg at the firing point, but was not detected in soil samples collected greater than 2 ft or in a down gradient monitoring well (AMEC 2002, 2000). Similarly, NG was detected in surface soil at two artillery/mortar firing points, as well as at Demolition Area 1 at Camp Edwards, but not in deeper soil samples (AMEC 2001a, 2001b) or in down gradient monitoring wells (AMEC, 2001c).

1.2 Dinitrotoluene

DNTs are nitro-aromatic compounds differing from 2,4,6-trinitrotoluene (TNT) only because they lack a third NO_2 group attached to the aromatic ring. The isomers of environmental importance are 2,4-DNT and 2,6-DNT. Both compounds are by products in the 2,4,6-trinitrotoluene (TNT) manufacturing process and are typically found in a 4:1 mass ratio of 2,4 to 2,6-DNT. In addition to their presence as manufacturing impurities, DNTs are used as a plasticizer, burn-rate modifier, and flash suppressant in gun propellants. Consequently, they have been identified at the SAR and gun and mortar firing points at Camp Edwards. DNTs have higher water solubilities (approximately 180 mg/L for 2,6-DNT and 270 mg/L for 2,4-DNT) than TNT (approximately 100 to 200 mg/L). DNT is susceptible to various transformation processes, and thus there are a variety of potential degradation pathways and potential daughter products (Thorn et al. 2008; Nishino et al. 2000). The common end product for these different pathways is nitrite-nitrogen, which tends to be unstable in soil and easily converted to ammonia-nitrogen.

Adsorption experiments were conducted specifically with Camp Edwards soils to determine the partitioning of 2,4-DNT for various experimental conditions and site soils (Speitel et al. 2002). The average K_d value for shallow soil was 3.3 L/kg, indicating somewhat greater adsorption to soil

than NG (Yamamoto et al. 2004; Speitel et al. 2002). In contrast, the only other study to evaluate the adsorption K_d of DNT assessed the relationship with varying clays (Haderlein et al. 1996). In that study, the K_d for 2,4-DNT mixed with kaolinite, illite, and montmorillonite was estimated at 690, 3,650, and 740 L/kg, respectively. Interestingly, the 2,6-DNT K_d values were much lower for kaolinite, illite, and montmorillonite, with values of 10, 52, and 125 L/kg, respectively.

1.3 Conceptual model

The fate-and-transport conceptual model for fired propellants begins with the deposition of gasses and particulates containing NG in a nitrocellulose binder (NC) and for some formulations containing DNT as part of the propellant grain and other materials onto the soil surface (Figure 1). The thickness of the arrow in Figure 1 reflects the expected importance of individual pathways, and a thicker line represents greater importance. NG can be deposited as a neat product (condensing vapor), but it is mostly likely deposited as a component within a NC matrix. Deposition likely consists of out gassing and particle deposition of uncombusted and combusted propellant residuals from the weapon barrel. Upon introduction to the environment, NC and NG undergo physical weathering, and the NG on the surface of the propellant fiber is dissolved in the presence of water whereas the balance of NG remains unexposed within the NC. NC is insoluble and poses a barrier that prevents precipitation from contact with the NG. Once NG is present in the dissolved phase, its fate-and-transport can proceed under a number of different pathways (Figure 1). Dr. Taylor at CRREL has evaluated the dissolution pathway (Appendix A). Although not studied in this project, phytoreduction and photo reduction may possibly also be active pathways for NG degradation. The literature suggests photo reduction may be an important process limiting movement of NG (Spanggord et al. 1980). This report focuses primarily on the adsorption/desorption pathway, and dissolution studies are attached as Appendix A. The last and likely most important mechanism is transformation of NG and DNT via abiotic and aerobic biodegradation processes. The dashed line (Figure 1) from the transformation pathway box to groundwater reflects the probability that this pathway is incomplete (i.e. the proceeding mechanisms tie up or transform any aqueous NG and DNT), preventing significant migration (i.e. more than a few feet from the soil surface). Evidence of daughter products is not anticipated due to equally significant degradation processes as noted for parent compounds.

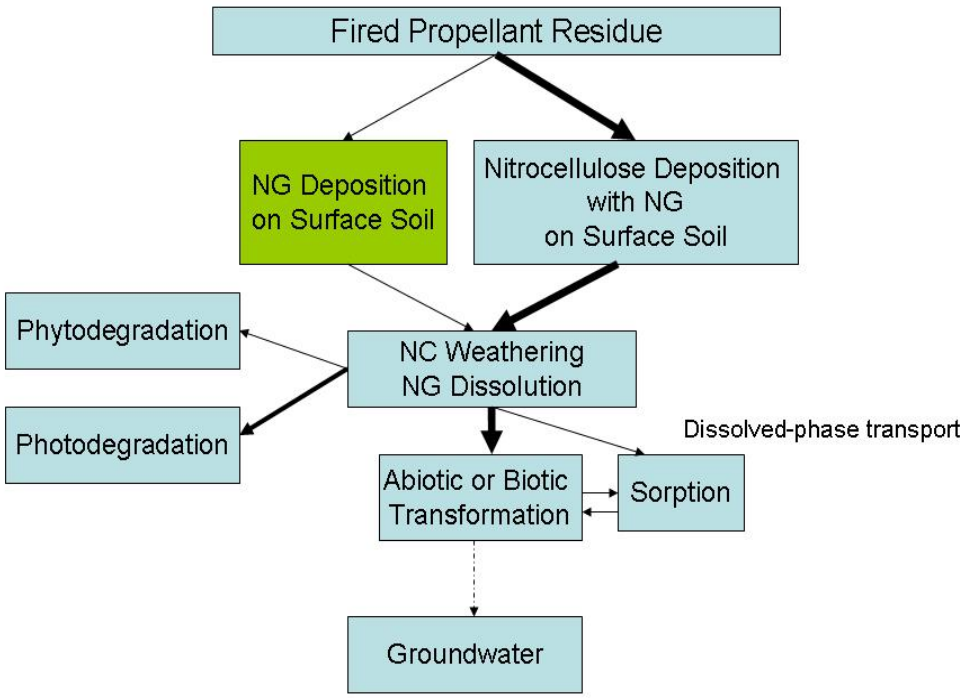

Figure 1. Fate-and-transport conceptual model for deposition of fired propellant containing NC, NG, and DNT.

2 Objectives

This study assessed the adsorption and desorption K_ds of dissolved NG and DNT with Camp Edwards soils. This information, plus the added steps of dissolution and degradation, may be utilized to develop loading rates for modeling the transport of these compounds from surface soil through the unsaturated zone. Column experiments typically yield a more representative K_d value than batch experiments and were conducted to augment the batch studies.

Drip experiments and standard batch tests are being carried out by Taylor at ERDC-CRREL to assess the dissolution of explosive and propellant powders in an ongoing research project funded by the Strategic Environmental Research and Development Program (SERDP) over the next 2 years. Taylor's work with NG and DNT from fired and unfired propellant used in SAR training is discussed in Appendix A.

A number of experiments were included to evaluate the potential impact of biodegradation by including batch and column tests without added biocide.

The project team for this work included the following personnel:

- U.S. Army Corps of Engineers, New England District (CENAE)
  Program Manager – Dave Margolis
- CENAE Technical Lead – Ian Osgerby
- CENAE Field Coordinator – John Macpherson
- ERDC-CRREL Principal Investigator – Jay Clausen
- ERDC-CRREL Laboratory Technician Constance Scott
- ERDC-CRREL Laboratory Technician Nate Mulherin
- ERDC-CRREL Laboratory Technician Susan Bigl

All laboratory studies related to this report were coordinated through Dr. Osgerby (CENAE), who was responsible for the execution of the project. Dr Osgerby reported progress and results to Mr. Margolis (CENAE). All field activities related to this project were coordinated through Mr.

Macpherson (CENAE). These activities included sampling of soils at three SARs (Echo, Juliet, and Kilo Ranges) at Camp Edwards to use in the experimental program as well as firing of small arms to allow collection of samples of fresh-fired propellant.

3 Methods

A series of batch tests were conducted for determining the adsorption and desorption K_ds of NG and DNT from the Camp Edwards SAR soils. Batch experiments involved spiking soil with the material of interest over a specified period of time. The K_ds are determined from the difference between the initial and final concentration of the aqueous solution, accounting for the volume of water and mass of soil used in the experiment. Advantages of batch experiments include rapid characterization of a variety of conditions, but the disadvantage is that these experiments do not reproduce the chemical reaction conditions in the natural environment. Desorption batch experiments were conducted because adsorption/desorption reactions are not always reversible. Typically, the adsorption process occurs more readily than the desorption process, resulting in higher desorption K_ds. Experiments were carried out principally with aqueous, reagent-grade NG and DNT with a few experiments assessing propellant-grade (unfired) materials, fresh-fired propellant materials, and a weathered, previously contaminated range soil.

The unfired propellant utilized was derived from the M855 projectile (5.56 mm) containing WC844 propellant. WC844 propellant is a mixture typically containing 66.95% NC, 11.2% NG, and 21.75% additives. The attached dissolution study report (Appendix A) includes photos and descriptions of the fired and unfired propellant grains. Individual, unfired propellant particles each contain a graphite coating, which increases its hydrophobicity. Fired grains are significantly different in appearance but remain similarly configured (Appendix A).

Contaminated soils containing NG and DNT from Camp Edwards were used to assess the desorption potential of weathered fired propellant, while fresh-fired propellant residues were obtained later and tested prior to the completion of the program.

3.1 Soil sample collection

Soil samples for the experiments were obtained from Echo (E), Juliet (J), and Kilo (K) SARs at Camp Edwards, Massachusetts (Figures 2 and 3). Six surface soil samples (0 - 3 in. below ground surface, two each per range), were collected using a systematic, nonrandom sampling technique utiliz

ing 100 increments (Hewitt et al. 2008). Sample collection occurred in an area presumed to be behind the firing point and thus free of propellant residues. Some of the soils contained detectable levels of NG and DNT. Consequently, additional soil samples were collected from an area of the SAR where propellant residues were later confirmed to be absent.

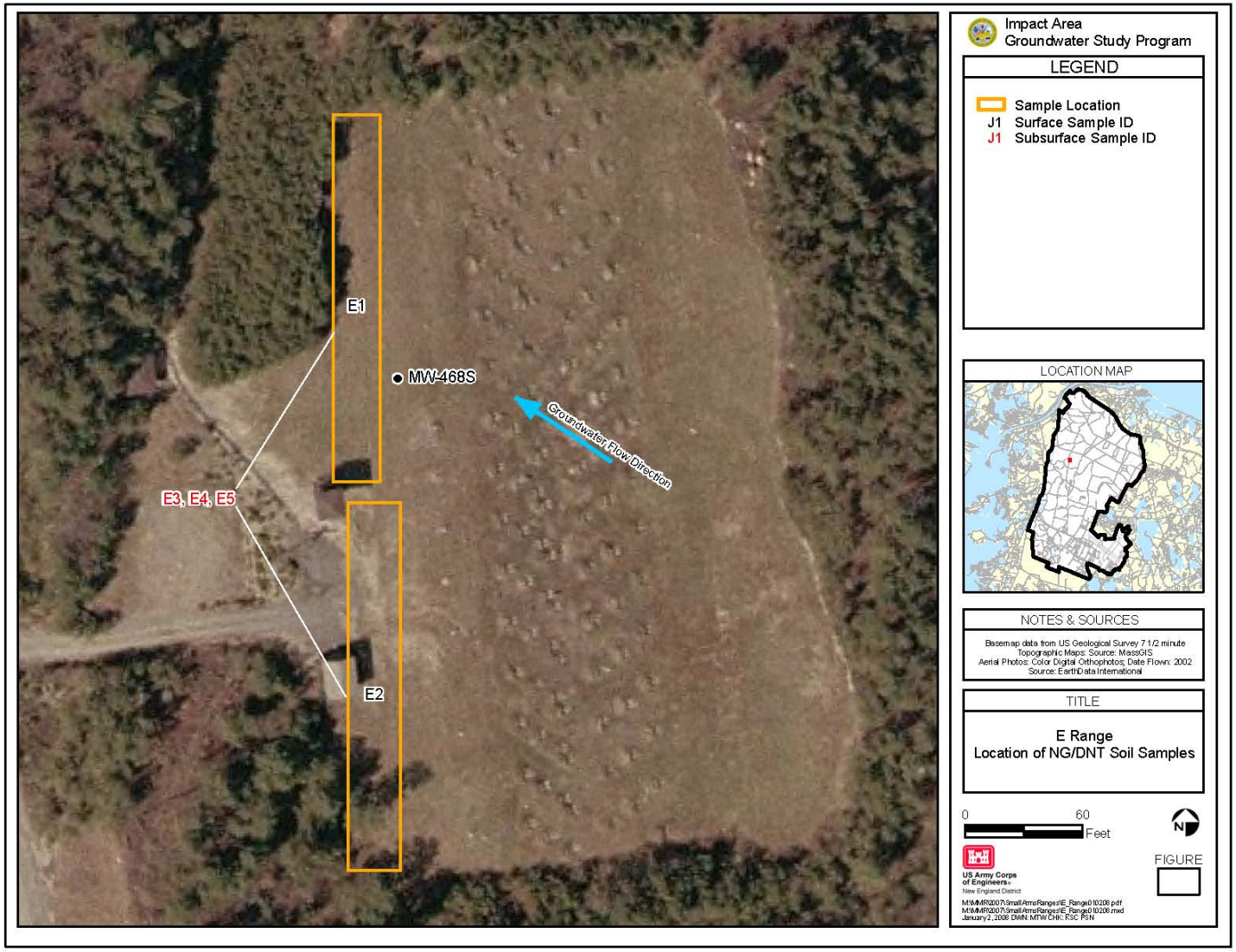

Figure 2. Location of sample collection at Echo Range.

Figure 3. Locations of sample collection at Juliet and Kilo Ranges.

Collecting 100 soil increments from the subsurface was impractical, and discrete samples are subject to biased heterogeneity or other differences in relation to those gathered at the surface. Therefore, subsurface soil samples were collected with a bucket auger from eight different locations per range, with those from the same depth interval combined to form a single 8-increment sample. Subsurface depth intervals assessed for each range were 9 to 12, 18 to 24, and 30 to 36 in.

Five soil samples were collected from Echo Range (E1 - E5) and seven each from Kilo (K1-K7) and Juliet (J1 - J7) Ranges (Figures 2 and 3). All soils designated with the number 1 and 2 were surface soils (0 - 3 in.) collected from behind what were believed to be firing points (Table 1). All soils with a 3 - 5 designation were subsurface soil samples (Table 1). Because NG and DNT were present at elevated levels in the J1, J2, K1, and K2 soils, four additional soil samples were collected downrange on the range floor near the berm and were designated J6, J7, K6, and K7. Since no contaminants (NG or DNT) were detected in the K7 surface soil and owing to its similar properties to the other soils (Table 2), K7 was selected as the default soil for all tests, except where soil heterogeneity was being evaluated, such as in Test 3. The Work Plan for USACE in 2007 called for using soil from Echo Range for the experiments; however, the presence of NG in the soils necessitated the need for collecting and using soil from an alternative location (Appendix B).

The following physical soil property data were collected to obtain site specific values; grain size distribution (i.e. percent sand, silt, and clay), bulk density, pH, fraction of organic carbon (OC), cation exchange capacity (CEC), extractable iron, and moisture content. Each of the soils was also analyzed for metal content (Table 2). Property data in Tables 1 and 2 do not indicate significant differences in the soils collected between Echo, Juliet, and Kilo Ranges. However, differences exist in the carbon content and CEC with depth for soils from all three ranges. Both OC and CEC decrease with increasing depth. In addition, the K6, K7, J6, and J7 soils have a higher lead content, presumably due to their closeness to the SAR impact berm. A slight decline in lead levels with increasing depth may also exist. The K6, K7, J6, and J7 soils contain tungsten, in contrast to the other soil samples without tungsten. The presence of tungsten is due to the closeness of the K6, K7, J6, and J7 samples to the SAR impact berm. The presence of lead or tungsten in the K6, K7, J6, or J7 was not expected to have an effect on the outcome of the adsorption or desorption tests. Adsorption sites for

ionic species are quite different than those for organic compounds, and no interference should occur.

Table 1. Summary of soil property information, including initial NG and DNT concentrations.

Range	ID	Date Collected	Location	Increments Collected	Depth (in.)	Moisture (%)	pH	Classification	2,4-DNT (mg/kg)	2,6-DNT (mg/kg)	NG (mg/kg)
Juliet	J1	11/8/2007	L side, behind FP*	100	0 - 3	13.20	5.92	Gravelly loamy sand	0.09	<0.018	3.00
Juliet	J2	11/8/2007	R side, behind FP	100	0 - 3	16.53	5.61	Gravelly loamy sand	0.08	<0.018	2.86
Juliet	J3	11/8/2007	n/a	8	9 - 12	12.46	6.30	Gravelly sandy loam	0.11	<0.018	3.35
Juliet	J4	11/8/2007	n/a	8	18 - 24	14.71	6.21	Gravelly sandy loam	<0.014	<0.018	0.21
Juliet	J5	11/8/2007	n/a	8	30 - 36	7.28	6.70	Gravelly sand	<0.014	<0.018	0.13
Juliet, East	J6	12/14/2007	3m from toe of berm	100	0 - 3	17.88	7.19	Gravelly sand	<0.014	<0.018	<0.020
Juliet, West	J7	12/14/2007	3m from toe of berm	100	0 - 3	17.62	7.18	Gravelly loamy sand	<0.014	<0.018	<0.020
Echo	E1	11/8/2007	L side, behind FP	100	0 - 3	11.68	6.82	Gravelly sand	<0.014	<0.018	0.36
Echo	E2	11/8/2007	R side, behind FB	100	0 - 3	11.15	6.44	Gravelly sand	<0.014	<0.018	0.43
Echo	E3	11/8/2007	n/a**	8	9 - 12	7.85	6.18	Gravelly sand	<0.014	<0.018	0.06
Echo	E4	11/8/2007	n/a	8	18 - 24	6.68	6.69	Gravelly loamy sand	<0.014	<0.018	<0.020
Echo	E5	11/8/2007	n/a	7	30 - 36	7.15	6.42	Gravelly loamy sand	<0.014	<0.018	<0.020
Kilo	K1	11/8/2007	L side, behind FP	100	0 - 3	18.51	5.57	Gravelly loamy sand	1.51	<0.018	69.6
Kilo	K2	11/8/2007	R side, behind FP	100	0 - 3	16.50	5.37	Gravelly loamy sand	1.14	<0.018	41.5
Kilo	K3	11/8/2007	n/a	8	9 - 12	16.29	5.72	Gravelly loamy sand	0.05	<0.018	1.44
Kilo	K4	11/8/2007	n/a	8	18 - 24	16.53	5.84	Gravelly loamy sand	<0.014	<0.018	0.69
Kilo	K5	11/8/2007	n/a	8	30 - 36	12.34	6.25	Gravelly loamy sand	<0.014	<0.018	0.67
Kilo, West	K6	12/14/2007	3 m south of berm	100	0 - 3	19.71	7.28	Gravelly loamy sand	<0.014	<0.018	<0.020
Kilo, East	K7	12/14/2007	3 m from toe of berm	100	0 - 3	20.96	7.28	Gravelly sand	<0.014	<0.018	<0.020

* FP – firing point; ** n/a – not applicable.

Sample Used for Adsorption Tests 1 – 6 and Desorption Tests 1 – 6

Mean of 3 replicates

Table 2. Summary of soil composition/property information.

		Total OC %	Inorganic Carbon %	Total Carbon %	Bulk Den-sity g/cc	CEC meq/ 100g	Percent Solids %	Metal Concentration mg/kg																					
Range	ID							Ag	Al	As	Ba	Be	Ca	Cd	Cr	Cu	Fe	K	Mg	Mn	Na	Ni	Pb	Sb	Se	Tl	V	Zn	W
Echo	E1	1.08	ND*	1.12	1.25	7.5	99.6	ND	4750	ND	13	0.22	1840	ND	690	21	11400	590	570	100	110	9.7	120	1.2	0.8	0.6	18	13	ND
Echo	E2	0.93	ND	0.95	1.25	7.7	99.7	ND	5930	ND	21	0.34	770	ND	630	8.3	13100	900	1060	130	120	10.2	17	ND	ND	ND	20	16	ND
Echo	E3	0.43	ND	0.43	1.3	4.9	99.7	ND	4960	ND	14	0.21	270	ND	580	19	10300	620	610	83	100	7.3	28	ND	ND	ND	16	7.9	ND
Echo	E4	0.22	ND	0.22	1.27	3.7	99.9	ND	3240	ND	13	ND	270	ND	640	ND	10400	640	400	77	120	8.0	2.7	ND	ND	ND	15	6.4	ND
Echo	E5	0.26	ND	0.28	1.3	3.3	99.9	ND	2600	ND	12	ND	250	ND	640	ND	9900	600	380	82	110	6.3	2.2	ND	ND	ND	12	6.0	ND
Juliet	J1	NA**	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Juliet	J2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Juliet	J3	0.39	ND	0.43	1.28	7.5	99.6	ND	11600	ND	28	0.45	410	ND	490	ND	15700	1210	1430	130	170	9.3	8.5	ND	ND	ND	23	33	ND
Juliet	J4	0.22	ND	0.22	1.23	8.6	99.5	ND	15500	ND	37	0.74	480	ND	340	5.2	18400	1810	2520	160	230	12.5	8.4	ND	ND	ND	30	24	ND
Juliet	J5	0.05	ND	0.09	1.31	3.0	99.9	ND	4260	ND	16	0.28	260	ND	580	ND	12600	790	800	140	110	7.8	24	ND	ND	ND	16	10	ND
Juliet, East	J6	1.44	ND	1.44	1.19	11.7	99.5	ND	6190	ND	18	0.25	3290	ND	670	73	12800	810	790	97	130	9.4	310	ND	ND	ND	19	17	103
Juliet, West	J7	1.40	ND	1.40	1.17	11.6	99.4	ND	6550	ND	19	0.28	3230	ND	640	60	12900	880	840	100	110	8.5	200	ND	ND	ND	18	18	29
Kilo	K1	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Kilo	K2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Kilo	K3	0.54	ND	0.57	1.26	11.1	99.3	ND	17800	ND	34	0.68	390	ND	370	ND	19600	1470	2280	140	180	11.3	9.2	ND	ND	ND	32	21	ND
Kilo	K4	0.27	ND	0.29	1.31	9.4	99.4	ND	17300	ND	35	0.68	410	ND	320	2.7	19700	1590	2430	140	200	11.8	7.9	ND	ND	ND	32	21	ND
Kilo	K5	0.16	ND	0.16	1.25	7.0	99.6	ND	13800	ND	34	0.70	440	ND	380	5.1	18200	1620	2260	160	210	11.3	7.0	ND	ND	ND	29	21	ND
Kilo, West	K6	2.22	ND	2.22	1.3	15.7	99.2	ND	7260	ND	22	0.33	15900	ND	600	390	13300	1100	1300	120	130	10.3	640	ND	ND	ND	21	64	52
Kilo, East	K7	2.72	0.07	2.79	1.22	17.0	99.0	ND	7550	ND	22	0.33	15400	ND	600	240	13300	1100	1480	120	120	9.1	700	ND	ND	ND	20	38	28

* ND – not detected; ** NA – not analyzed.

Sample Used for Adsorption Tests 1 – 6 and Desorption Tests 1 – 6.

Mean of 3 replicates.

Soil was used from each of the three ranges and from each of the four depths for the batch tests. Kilo Range soil K7 was used as the control soil for the majority of the tests where a single variable was being changed. The Work Plan (USACE, 2007) had called for using soil from Echo Range for the batch tests but the presence of propellant residues in these samples prevented their use (Appendix B). The size and layout of Echo Range prevented the collection of additional samples. Soil samples for the column experiments consisted of a mixture of K6 and K7 soils (surface soil collected downrange on Kilo Range). The K6 and K7 soils were mixed together to provide enough soil for all eight tested columns (four separate individual tests with four replicates).

3.2 Analytical methods

Analysis of NG, DNT, and GDN compounds for all experiments generally followed USEPA Method 8330B (USEPA 2006), using high performance liquid chromatography (HPLC). A subset of samples was analyzed using a gas chromotgraphy – electron capture detector (GC-ECD) generally following EPA Method 8095 for confirmation (USEPA 1998). Because the analytes of interest were known, dual-column confirmation was not utilized for either method. Chloride samples were analyzed by ion chromatography, and metals analysis was performed by inductively-coupled mass spectrophotometry.

3.2.1 High performance liquid chromatography

The sample preparation and analytical procedures for the batch and column experiments were as follows. Batch test aqueous samples were allowed to settle for 2 hr prior to transferring 1 mL of solution to a clean (washed with deionized water (DI)) 40 mL-amber glass vial containing 29 mL of autoclaved, organic-free, DI and 10 mL of acetonitrile. Column test aqueous samples were ready for preparation upon collection. The transferred solution was then filtered through a 0.45-µm Millex FH filter and transferred to a 7-mL amber glass vial. A 2-mL subsample was then placed in an auto sampler vial for analysis.

If analysis within 24 hr was not possible, the samples were chilled to 4°C until analysis was performed following CRREL procedures. As discussed in Section 5, freezing of samples was not necessary, and chilling was sufficient to prevent losses. Although freezing was called for in the Work Plan (USACE 2007) the elimination of the thawing cycle associated with freez-

ing allowed for a more efficient test procedure and eliminated concern about thawing samples at room temperature for an extended period of time (Appendix B).

Soil samples from the batch or column experiment were analyzed by placing a 10-g aliquot of soil and 20 mL of acetonitrile in an amber glass jar, followed by 18-hr of extraction on a table top shaker. Since the majority of experiments consisted of the application of an aqueous mixture of NG and DNT, these samples were not pulverized prior to analysis as proscribed in Method 8330B. For those samples containing unfired (Test 7d) or fired propellant (Test 10d), the entire soil sample was extracted with acetonitrile. Complete sample extraction was performed for the 0 - 2 cm layer in Column 3B where fired propellant residue was placed on the soil surface. A complete soil extraction was performed since the sample mass in the experiment (14 g for the batch experiment, 30 g for the column) was too small to be ground. The grinder apparatus at CRREL requires a minimum sample mass of 500 g. Soil samples collected from the columns at a depth greater than 2 cm were not ground since particulate migration was not anticipated, and the recovered soil sample mass was too small for grinding. After extraction, the sample was allowed to settle for 2 hr. An aliquot of each extract was then passed through a 0.45-µm Millex FH filter and transferred to a 7-mL amber glass vial. The final preparation step prior to HPLC analysis was to mix one part of the acetonitrile extract with three parts autoclaved, reagent-grade DI. A 2-mL sub sample was then placed in an auto sampler vial for analysis.

Analysis using reversed-phase HPLC-ultraviolet was performed following USEPA Method 8330 (USEPA 1994), using a Thermo Finnigan system comprised of a Spectra System Model P1000 isocratic pump, a Model AS300 auto sampler, and a Model UV2000 dual-wavelength absorbance detector set at 210 and 254 nm. The HPLC separations were performed using a 15-cm × 3.9-mm (4-µm) Nova Pac C_8 (Waters Millipore) column at 28°C eluted with a 15:85 isopropanol:water mix at 1.4 mL/min. A 1.0 mg/L calibration standard was run for every 10 samples. Many of the samples were analyzed twice for confirmation purposes. Estimated reporting limits and method detection limits are provided in Table 3. Aqueous samples were not pre-concentrated using solid phase extraction (SPE) due to the elevated NG and DNT concentrations utilized for the experiments. Additional discussion on quality assurance and quality control is provided in Appendix E.

3.2.2 Gas chromatography

To confirm the presence of NG, DNT, and GDN, a second analysis was conducted on a subset of sample extracts, including all those with low concentrations of energetic compounds, using GC-ECD. Lower estimated reporting limits are possible since the GC-ECD has greater sensitivity than the HPLC. The purpose for analysis on the GC-ECD was to provide confirmation of low levels of NG and DNT. A second purpose was to provide confirmation of potential detections of 1,2-GDN and 1,3-GDN.

Table 3. Method detection limit (MDL) and estimated reporting limit (ERL) for HPLC and GC-ECD analyses of aqueous and soil samples in this study.

	Concentration		
Method / Limit	2,4-DNT	2,6-DNT	NG*
HPLC Aqueous (mg/L)			
ERL	0.10	0.20	0.25
MDL	0.02	0.04	0.05
HPLC Soil (mg/kg)			
ERL	0.031	0.049	0.065
MDL	0.0062	0.0098	0.013
GC-ECD Aqueous (µg/L)			
ERL	0.01	0.01	0.20
MDL	0.002	0.002	0.04
* Limits for 1,2-GDN and 1,3-GDN are estimated to be the same as for NG. Note: Quantification between MDL and ERL are reported as J values.			

Water samples for GC analysis were pre-concentrated by passing through a SPE cartridge (Jenkins et al. 1995). This technique retains the energetic residues on a Porapak RDX cartridge (Sep-Pak, 6 cm^3, 500 mg, Water Corporation) that was subsequently eluted with 5.00 mL of acetonitrile. Direct injection of 1 µL of the eluent was made into a purged-packed injection port (250°C) equipped with a Restek Direct Injection Uniliner. These analyses were conducted on an HP 6890 Gas Chromatograph equipped with a micro ECD detector. Primary separation was conducted on a 6-m- × 0.53-mm-ID fused-silica column, with a 1.5-µm coating of a proprietary phase (Rtx-TNT from Restek, Bellefonte, Pennsylvania). The GC oven was temperature programmed as follows: 100°C for 2 min, 10°C/min ramp to 180°C, 30°C/min ramp to 300°C and held for 8 min. The carrier gas was hydrogen flowing at a constant 13.6 mL/min over the temperature-

programmed analysis run time. The ECD detector temperature was 310°C, and the makeup gas was nitrogen flowing at a constant 45 mL/min. Multi-analyte standards were purchased from Restek, and the instrument was calibrated over three concentrations between 100 and 800 μg/L.

3.2.3 Ion Chromatography

Anion concentrations, principally chloride, were measured on a Dionex ICS-3000 ion chromatograph using an AS-19 anion column with a 10 μL injection volume. A gradient method using a KOH eluent concentration ranged from 20 mM to 35 mM. The flow rate was constant at 1 mL/min and the operating temperature was 30˚C. The ion chromatograph was calibrated using standards with a range of values from 0.5 to 28 mg/L. Multiple analyses of calibration standards, sample duplicates, and a 50 mg/L sodium chloride standard solution yielded a calculated precision for the analyses of = +/- 2%. A solution of 18.2-MΩ water was routinely analyzed, and no peaks were obtained. Peaks were identified for the column samples using Chromeleon (Dionex), and each peak was visually investigated.

3.2.4 Inductively-coupled optical emission spectrometer

Inductively-Coupled Mass Spectrometry (ICP-OES) was used to obtain the metal (Ag, Al, As, Ba, Be, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, Se, Tl, V, Zn, and W) content of the soil samples. Sample digestions using microwave heating were performed following EPA SW-846, Method 3051 (USEPA, 1996). The soil samples were analyzed at ERDC-EL using a Perkin Elmer Optima 4300 DV ICP-OES following EPA Method SW-846 Method 6010A (USEPA 1996).

3.2.5 Other methods

Laboratory measurements of water samples for pH and temperature were obtained with a Model 556 MPS YSI meter. Organic carbon was measured by USEPA Method 9060, and cation exchange capacity was measured by USEPA Method 9081. The bulk density of soil was measured following ASTM Method D6683-01.

4 Batch Experiments

Data reduction and example calculati0ns are outlined in Appendix C. All of the HPLC data for the batch adsorption and desorption tests are provided in Appendix F, with K_d calculation results provided in Appendix G. The estimated soil concentrations at the end of the adsorption and desorption tests are provided in Appendix H.

4.1 Adsorption experiment procedures

Adsorption behavior of NG and DNT was explored with laboratory batch studies to quantify the interaction with clean-site surface and subsurface soils. The purpose of these experiments was to evaluate how well these compounds introduced as a solution bind to site soils. This information in combination with the measured equilibrium liquid concentration, soil mass, and liquid volume can be used in Equation 2 (Appendix C) to develop an adsorption K_d for NG and DNT. A range of soil conditions, such as different samples from within a SAR, different SARs, depth, time, temperature, pH, and concentration were evaluated. The concentration of the reagent-grade, aqueous spiked solution for the adsorption tests was the same for all experiments (10 mg/L of 2,4-DNT, 2,6-DNT, and NG), except for one test series (Test 2) where varying concentrations were assessed. Use of an aqueous-spiked solution eliminates the possible effects of dissolution. The same surface soil (K7) was used for all of the adsorption experiments, where a single variable was changed (i.e. concentration, time, temperature, or pH). A biocide consisting of mercuric chloride (0.02%) and glutaraldehyde (1%) was added to all samples except for some of the Test 8, 9, 10, and 11 experiments. Biocide greatly reduces biological activity within the soil. All experiments were conducted in amber 4-oz glass jars to eliminate the possibility of photo degradation. Combined with the use of the aqueous spiked solution, the biocide allows for a strict focus on adsorption. Additionally, glass was selected because the medical industry has shown that NG can stick to plastics. Because DNT volatility is low but not zero, Teflon lids were used to prevent any volatile DNT loss.

Adsorption experiments generally followed the guidelines of ASTM 4646-03 (ASTM 2004). Each experiment was performed in triplicate. In some cases, duplicate analysis was performed on the sample aliquot obtained from each experiment. Variables (Table 4) examined include:

- Time – five exposure periods
- Concentration – five different solution concentrations
- Inter-site heterogeneity – three different SARs (Echo, Juliet, and Kilo Ranges)
- Intra-site heterogeneity – six different soil locations (two locations per range)
- Depth – four different soil sample depths (0-3, 9-12, 18-24, 30-36 in.)
- Temperature – three different levels
- pH – three different levels

Table 4. Adsorption test experimental design.

Experiment	Inter-Site Surface Heterogeneity	Intra-Site Surface Heterogeneity	Depth	Concentration	Time	Temp	pH	Other	Replicate	Total Samples
Pre-Test 1 – Sample Storage-	1	1	1	1	1	1	1	17	1	17
Pre-Test 2 – Biocide Type Assessment	1	1	1	1	1	1	1	3	3	9
Pre-Test 3 – Biocide Time Assessment	1	1	1	1	5	1	1	1	1	5
Pre-Test 4 – Soil to Solution Ratio	1	1	1	1	1	1	1	3	3	9
DI Control Part of All Tests	1	1	1	1		1	1	6	1	6
Killed Soil Control Part of All Tests	1	1	1	1	1	1	1	6	1	6
Test 1 – Equilibration Time	1	1	1	1	5	1	1		3	15
Test 2 - Concentration	1	1	1	5	1	1	1		3	15
Test 3 - Heterogeneity	3	6	1	1	1	1	1		3	54
Test 4 – Soil Depth	1	1	3	1	1	1	1		3	9
Test 5 - Temperature	1	1	1	1	1	3	1		3	9
Test 6 - pH	1	1	1	1	1	1	3		3	9
Test 9 - Rainwater	1	1	1	1	1	1	1	4	3	12
Total										175

Batch-type equilibration tests were carried out using approximately 14 g of uncontaminated air-dried soil placed in a 4-oz amber glass jar with a Teflon lid. Soil was mixed with approximately 70 mL of autoclaved, organic-free DI containing approximately 10 mg/L of reagent-grade NG, 2,4-DNT, and 2,6-DNT, yielding a soil to solution ratio of 1:5. The Work Plan (USACE 2007) called for a soil to solution ratio of 1:20, assuming liquid phase concentration decreased by 50% during each test (Appendix B). However, preliminary experiments did not indicate 50% loss at the 1:20 ratio. A concentration decrease of 50% was targeted to ensure the analytical error inherent in the measurement of the initial and final liquid phase concentrations was not the controlling factor in estimating K_ds. Conse-

quently, a larger mass of soil was used in the batch experiments than specified in the Work Plan (USACE 2007).

Soil was homogenized by manual mixing prior to being added to the sample jars. A solution consisting of autoclaved, organic-free DI mixed with reagent-grade NG and DNT (obtained from Restek Inc.) was prepared. For all batch experiments, reactor vessels were shaken with an orbital shaker at approximately 150 revolutions per minute at room temperature (25°C); two experiments conducted at a lower (12°C) and a higher (32°C) temperature to illustrate any temperature dependence were not shaken. After the equilibration time, the soil-solution slurry was allowed to settle for 2 hr in the original bottles.

Each test was conducted in triplicate with at least one procedural blank. Control experiment jars containing spiked solution without soil were used to assess losses through adsorption to the glass, or reactions within the jars, and to confirm starting concentrations. Batch samples generated in the adsorption tests were subsequently used for the batch desorption experiments.

4.2 Desorption experiments procedures

The purpose of the desorption experiments is to assess whether adsorption is a linear or non-linear reversible process for the Camp Edwards soils. If adsorption is completely reversible, derived K_ds for the adsorption test will be valid for NG and DNT desorbing from soil over time. However, NG in propellant grains is mixed with NC and other stabilizers, and only a small portion of NG is located on the surface of the propellant grain. The propellant mixture is covered by a graphite coating, which further affects the release rate of NG. Therefore, desorption values obtained with aqueous, reagent-grade compounds adsorbed onto soil do not represent mechanisms occurring in surface soil at a SAR because there are dissolution/adsorption/desorption/diffusion/weathering/degradation process components involved with the solid propellant containing NC and NG in the field. Initial tests with reagent-grade NG and DNT were supplemented with samples of contaminated soil containing weathered, fired propellant residues, and later with freshly-fired and unfired propellant. Tests were conducted with and without biocide. Propellant and biocide were added to uncontaminated K7 soil. With knowledge of the desorption K_d, and elimination of other fate-and-transport processes such as degradation, it may

be possible to elucidate the dissolution component of the tests with fired and unfired propellant.

Desorption batch experiments were conducted in the same manner as the adsorption experiments, assessed the same variables (equilibration time, inter-site surface heterogeneity, intra-site surface soil heterogeneity, depth, concentration, time, temperature, rainwater, and pH), and were carried out using uncontaminated DI. The essential difference is that these tests started with contaminated soil generated during the adsorption tests. The same batch reactors used in the adsorption tests were used for the desorption tests, repeating the same experimental protocols (Table 5).

Soil was previously prepared for the adsorption test by adding 70 mL of DI containing 10 mg/L each of reagent-grade NG, 2,4-DNT, and 2,6-DNT to uncontaminated soil (K7) and equilibrating for 24 hr. The lag

Table 5. Desorption test experimental design.

Experiment	Inter-Site Surface Heterogeneity	Intra-Site Surface Heterogeneity	Depth	Concen–tration	Time	Temp	pH	Other	Replicate	Total Samples
Pre-Test 2d - Biocide Type Assessment	1	1	1	1	1	1	1	4	3	12
DI Control Part of All Tests	1	1	1	1	1	1	1	10	1	10
Killed Soil Control Part of All Tests	1	1	1	1	1	1	1	10	1	10
Test 1d- Equilibration Time	1	1	1	1	5	1	1	1	3	15
Test 2d - Concentration	1	1	1	5	1	1	1	1	3	15
Test 3d - Heterogeneity	3	2	1	1	1	1	1	1	3	18
Test 4d – Soil Depth	3	1	3	1	1	1	1	1	3	27
Test 5d - Temperature	1	1	1	1	1	3	1	1	3	9
Test 6d - pH	1	1	1	1	1	1	3	1	3	9
Test 7d- Unfired Propellant	1	1	1	1	1	1	1	2	3	6
Test 8d – Contaminated Soil	1	2	1	1	1	1	1	2	3	12
Test 9d - Rainwater	1	1	1	1	1	1	1	4	3	12

Test 10d – Fired Propellant	1	1	1	1	1	1	2	2	3	12
Total										**167**

between the adsorption and desorption tests varied from 1 week up to several weeks. The Work Plan (USACE 2007) called for an arbitrary minimum 2-week interval between the adsorption and desorption experiments. However, initial project delays resulting from the need to resample the ranges due to the presence of propellant residue and the requirement for meeting a scheduled Interim Batch Test Report necessitated some compression of the experiment schedule (Appendix B). Therefore, in some cases it was necessary to have less than 2 weeks between the adsorption and desorption experiments. The soil from the adsorption tests was allowed to air dry in the 4-oz amber jars for a minimum of 24 hr and then weighed. To begin the desorption test, 70 mL of DI was added to the soil. Samples were then equilibrated for 24 hr on an orbital shaker table. After 24 hr the desorption test samples were allowed to settle for 2 hr, and the aqueous solution was prepared for analysis using the same procedures, methods, and equipment utilized for the batch adsorption tests.

In addition to the seven desorption tests that mimicked the adsorption tests, three supplemental tests, Desorption Tests 7, 8, and 10 were conducted to assess the adsorption/desorption characteristics of unfired military propellant in K7 soil, contaminated Kilo Range (K-1/K-2) surface soils containing weathered propellant, and freshly-fired propellant added to the K7 soil. The tests should not be considered strictly desorption tests because the propellant residue first has to dissolve and interact with the soil and then be desorbed from the soil. As noted in this report, weathering is a significant process at SARs because available propellant from freshly-fired ammunition is readily leached out onto the soil, and the residual propellant remains encapsulated in the hydrophobic NC. This encapsulated propellant appears to be essentially immobile and unavailable. In Tests 7d and 10d, solid unfired and freshly-fired propellant was mixed with uncontaminated soil (K7) to achieve a total (but not necessarily available) NG soil concentration of approximately 1,120 mg/kg. Test 9d was conducted to evaluate the difference between DI and uncontaminated rainwater for the K7 soil.

For Batch Test 10d, as well as Columns 3A, 3B, 4A, and 4B, fired propellant was obtained from a number of rounds fired through a plastic lined enclosure (Fig. 4). A total of 180 of 5.56-mm rounds were fired, as well as

100 of 7.62-mm and 150 of 9-mm rounds (Table 6). The residue was swept up off the plastic and placed into amber glass jars. Because very little residue was collected from the 7.62-mm ammunition, when experiments utilized fired propellant, a mixture of equal parts 9-mm and 5.56-mm residue was used.

Figure 4. Residue collection chamber.

Table 6. Information for recovered propellant residue firing activity.

Ammunition DODIC Number	Type of Munition	Number of Rounds Fired	Possible propellant ID	Total Unfired Propellant Weight (g)	Fired Propellant Mass Recovered (g)	% Recovered	% NC	% NG	Total NG Mass in Recovered Propellant (g)
A059	5.56 mm	180	WC844	286.23	2.32	<0.008	72.15	11.0	0.3
A762	7.62 mm	100	WC867	3758.34	0.005	negligible	85.40	7.0	negligible
A363	9 mm	150	HPC33	50.54	3.09	0.06	78.09	9.5	0.3

4.3 Preliminary tests

A series of preliminary experiments was conducted to evaluate storage stability issues, performance of selected biocides, biocide contact time, and the ideal soil-to-solution ratio (Table 7). In the first set of experiments, Pre-Test 1, a water sample was spiked with reagent-grade DNT and NG and either frozen at -18°C or refrigerated at 0°C. Figure 5 indicates there was no loss of DNT or NG over a 28-day period. Therefore, storage of test

aliquots prior to analysis is not an issue. Although the Work Plan (USACE, 2007) called for freezing of samples prior to analysis, an extended thawing period would have been necessary and potentially affected the results (Appendix B). Chilling also allowed for improved efficiency during the sample preparation stage. A spiking solution was prepared for each batch experiment with an associated control sample.

Table 7. Comparison of glutaraldehyde versus mercuric chloride impact on partitioning coefficients.

Pre-Test 3 - Evaluation of Adsorption K_d by Biocide Type					Pre-Test 3d - Evaluation of Desorption K_d by Biocide Type				
		K_d (L/kg)					K_d (L/kg)		
Biocide Type	Rep	2,4-DNT	2,6-DNT	NG	Biocide Type	Rep	2,4-DNT	2,6-DNT	NG
Glutaraldehyde	1	1.3	1.7	0.6	Glutaraldehyde	1	7.1	10.5	6.1
	2	0.9	1.4	0.6		2	5.6	9.4	6.4
	3	1.1	1.5	0.6		3	6.7	10.0	6.3
	Mean	1.1	1.5	0.6		Mean	6.4	10.0	6.2
Mercuric Chloride	1	1.6	1.9	0.6	Mercuric Chloride	1	10.8	11.8	6.8
	2	1.2	1.7	0.5		2	8.6	10.9	6.7
	3	1.4	2.0	0.6		3	9.8	12.0	6.9
	Mean	1.4	1.9	0.6		Mean	9.7	11.6	6.8
Test used 14 g of K7 soil ,70 mL of DI, and added biocide..					Test used 14 g of K7 soil, 70 mL of DI, and added biocide.				

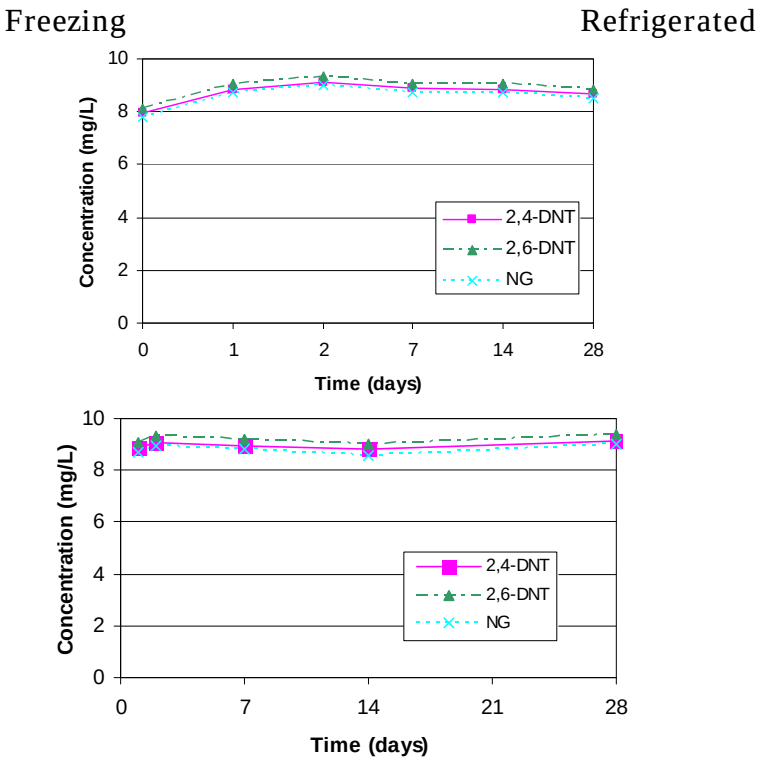

Figure 5. Comparison of freezing and refrigerated NG and DNT spiked water samples over 28 days.

A second set of preliminary experiments, Pre-Test 2, was conducted to assess the utility of mercuric chloride and glutaraldehyde as biocides. A 100 mg/L stock solution was diluted with biocide and DI to result in a final spike solution at 10 mg/L concentration of NG and DNT. No difference was evident in NG or DNT concentration levels between samples with biocide and those without biocide (Figure 6). Similar experiments were then conducted with the K7 soil. A 70-mL solution of DI with 10 mg/L reagent-grade DNT and NG was then added to 14 g of K7 soil. One set of samples had no biocide, and another set had added mercuric chloride (0.02%) and glutaraldehyde (1%). Slight differences were apparent between those soils inoculated with biocide and those without biocide (Figure 7). The results suggested a 0.5 mg/L loss for the 2,4 DNT samples without biocide when compared to those with biocide and a 1.0 mg/L loss for NG samples without biocide when compared to those with biocide. A slight and possibly anomalous increase is noted for 2,6 DNT between samples with or without biocide. The losses for those soils with no biocide suggest that microbiological activity is potentially responsible. The results indicate that the selection of biocides was generally effective for limiting microbiological activity in soil samples and was used in all subsequent tests. The differences

in starting and ending concentration between 2,4-DNT, 2,6-DNT, and NG in Figure 7 is presumed to be a function of the difference in the degree of adsorption to the soil.

One apparent analytical issue was that biocide appeared to elute at the same time as 2,4- and 2,6-DNT. CRREL's procedure for aqueous extraction involves 10 mL of sample added to 20 mL of DI and 10 mL of acetonitrile. This mixture was modified to 1 mL of sample, 29 mL of DI, and 10 mL of acetonitrile for the adsorption tests, which diluted the glutaraldehyde sufficiently to minimize its interference with the analytes of interest and maintained the anticipated concentration within the calibration range. The 0.1 and 1 mg/L spiked adsorption test and desorption test samples for batch test 1 were prepared for HPLC analysis by using 10 mL of sample with 20 mL of DI and 10 mL of acetonitrile.

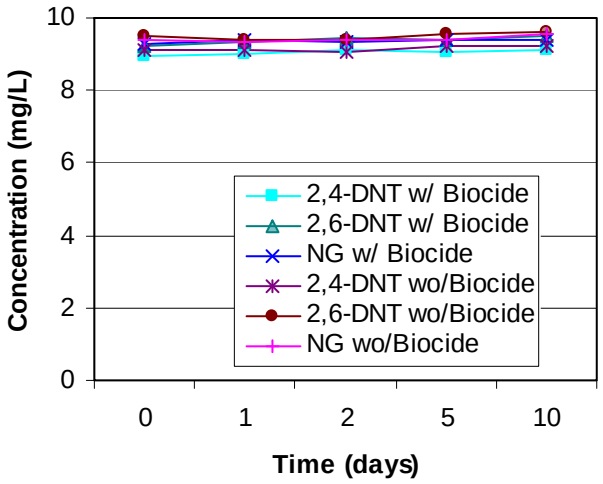

Figure 6. Comparison of NG and DNT levels for water with and without biocide added over 10 days.

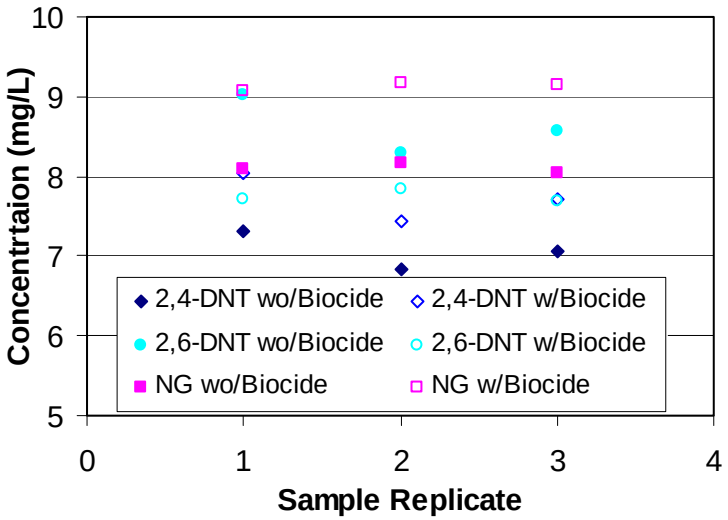

Figure 7. Comparison of NG and DNT levels in the aqueous phase after addition of reagent-grade NG and DNT with and without biocide to K7 soil.

In addition to these tests, biocides were evaluated individually with K7 soil (Table 7), with no significant difference in biocide effectiveness based on the adsorption and desorption K_d calculations.

A fourth set of preliminary experiments (Pre-Test 4) explored the ideal soil-to-solution ratio. The Work Plan (USACE 2007) called for using a 1:20 ratio with the intent of achieving 50% adsorption of the NG/DNT aqueous phase onto the soil (Appendix B). Initial experiments indicated approximately 20% of the NG/DNT was adsorbed onto the soil when using a 1:20 soil:solution ratio. To achieve a higher adsorption rate the soil:solution ratio was changed to 1:5, which resulted in approximately 30 to 50% adsorption of the NG/DNT onto the soil. Consequently, all subsequent tests used a 1:5 soil:solution ratio consisting of 70 mL of DI spiked with aqueous, reagent-grade NG and DNT, which was then added to 14 g of soil in each test vial.

4.4 Test 1 – Equilibration time

The Test 1 batch adsorption experiment evaluated the impact of equilibration time on the resulting K_d value. Earlier work by Speitel et al. (2002) suggested an equilibration time longer than 24 hr, as called for in the ASTM 4606-03 "Standard Test Method for 24-h Batch-Type Measurement of Contaminant Sorption by Soils and Sediments" guidelines, may be necessary based on observations with hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) and HMX. Surface soil (K7) from the Kilo Range (0 to 3 in.) was

used in this experiment, and the test was conducted over a contact time up to 216 hr following the methodology outlined in Section 3. The Work Plan (USACE 2007) called for ending the experiment at 240 hr but, because this occurred over a weekend, the test was terminated a few hours earlier (Appendix B). The results for the adsorption experiment (Table 8) indicate no difference in K_d values over 216 hr.

In addition, the equilibration time for the batch desorption test was investigated according to the methodology outlined in Section 4.2, and the results indicated no difference in K_d desorption over an interval of 240 hr for 2,4-DNT (Table 8). The experimental error (standard deviation) for the desorption tests is small and less than 0.4 L/kg for the calculated desorption K_d.

Desorption K_ds for DNT are slightly higher than those obtained during adsorption tests. This finding is consistent with observations with most organic compounds. When a chemical is sorbed to the soil, the energy to break the bonds between the soil and organic compound is greater and less material is desorbed resulting in higher desorption K_d values. The NG K_d for desorption indicates more variability as shown by the number of experiments where the calculated K_d value is zero (Table 8). For statistical purposes, a calculated negative K_d number was assigned a value of zero. However, this may be attributable to experimental/analytical errors due to the small differences in the small involved quantities. This variability reflects some of the difficulties in computing small changes in small numbers. Variability could also indicate that desorption experiments may not have achieved equilibrium at 24 or even 48 hr. The value at 72 hours suggests equilibrium was achieved. Then again, the results at 120 hr suggest a possible departure from equilibrium. Since different aliquots of soils were used in these tests it may also simply reflect variability in soil properties. Nevertheless, these NG results indicate all or most of the NG that sorbed onto the soil readily desorbed during the desorption test.

Table 8. Adsorption and desorption partitioning coefficients (K_d) by equilibration time for Tests 1 and 1d.

Test 1 - Evaluation of Adsorption K_d with Time					Test 1d - Evaluation of Desorption K_d with Time				
		K_d (L/kg)					K_d (L/kg)		
Time (hr)	Rep	2,4-DNT	2,6-DNT	NG	Time (hr)	Rep	2,4-DNT	2,6-DNT	NG
24	1	4.0	3.0	0.9	24	1	5.5	2.9	0.0
	2	4.0	2.9	1.0		2	5.4	3.0	0.0
	NA*	NA	NA	NA		2 dup**	5.4	3.0	0.0
	3	4.3	3.2	1.2		3	6.4	3.9	0.1
	NA	NA	NA	NA		1r	5.8	3.0	0.0

	NA	NA	NA	NA		2r	5.7	3.0	0.0
	NA	NA	NA	NA		2r dup	5.7	3.0	0.0
	NA	NA	NA	NA		3r	6.3	3.9	0.1
	Mean	4.1	3.0	1.0		Mean	5.8	3.2	0.03#
48	1	4.2	3.1	1.1	48	1	6.3	4.1	0.8
	1 dup	3.9	2.9	0.9		NA	NA	NA	NA
	2	4.0	2.7	1.1		2	5.7	3.1	1.0
	3	3.7	2.6	0.8		3	5.6	3.0	0.0
	NA	NA	NA	NA		1r †	6.6	4.2	0.9
	NA	NA	NA	NA		2r	5.9	3.1	1.0
	NA	NA	NA	NA		3r	5.9	3.0	0.0
	Mean	3.9	2.8	1.0		Mean	6.0	3.4	0.6#
72	1	4.2	3.2	1.1	72	1	6.5	5.3	2.1
	2	4.2	3.2	1.1		2	6.1	5.2	1.4
	3	4.0	3.0	1.0		3	5.5	4.7	1.8
	NA	NA	NA	NA		1r	6.6	5.4	2.1
	NA	NA	NA	NA		2r	6.2	5.4	1.5
	NA	NA	NA	NA		3r	5.6	4.8	1.9
	Mean	4.1	3.1	1.1		Mean	6.1	5.1	1.8
120	1	4.1	3.1	1.1	120	1	5.4	5.2	0.3
	2	4.2	3.0	1.0		2	5.7	5.0	0.3
	3	4.1	2.9	0.9		3	5.2	4.7	0.0
	NA	NA	NA	NA		1r	5.4	5.0	0.3
	NA	NA	NA	NA		2r	5.7	4.9	0.4
	NA	NA	NA	NA		3r	5.1	4.5	0.0
	Mean	4.1	3.0	1.0		Mean	5.4	4.9	0.2#
216	1	4.5	3.1	1.2	240	1	6.3	4.7	1.9
	1 dup	4.6	3.4	1.3		1 dup	NA	NA	NA
	2	4.5	3.3	1.3		2	6.1	5.0	1.7
	3	4.5	3.2	1.3		3	6.4	4.9	2.3
	Mean	4.5	3.3	1.3		Mean	6.3	4.9	2.0
Overall Mean		4.2	3.1	1.1	Overall Mean		5.9	4.2	0.8#
Standard Deviation		0.3	0.2	0.2	Standard Deviation		0.4	0.9	0.8#
All tests used 14 g of K7 soil with 70 mL DI spiked with 2,4-DNT = 9.11 mg/L, 2,6-DNT = 9.01 mg/L, and NG = 9.24 mg/L.					All tests used corresponding adsorption test soil with 70 mL of DI added.				
* NA – not applicable; ** dup – duplicate.					† r reverse order of analysis.				
# Calculated negative K_d values were assigned a value of zero for statistical calculations.									

Because of the compressed nature of the schedule, many desorption experiments were conducted in parallel. The results from adsorption tests indicated equilibrium had been achieved within 24 hr, and it was assumed a similar result would be obtained for desorption. As a consequence, all subsequent adsorption and desorption tests (Tests 2 through 10) utilized an equilibration interval of 24 hr. In retrospect, although a steady state ad-

sorption had been demonstrated, it is not unequivocally clear that equilibrium desorption was achieved in 24 hr for NG.

As noted in discussion of different test results, in some experiments desorption equilibration appears to have been achieved for NG in 24 hr, whereas in other experiments equilibrium may not have been achieved. This issue does not appear to be applicable to DNT, which is probably due to the greater degree of initial adsorption onto the soil. However, given the low NG K_d values for both adsorption and desorption, the uncertainty of the absolute NG desorption K_d value for some experiments does not effect the overall interpretation of the data. Data indicate the values of the K_d for NG are small (< 3), essentially reversible, and consequently applicable for modeling purposes where only simple equilibrium partition coefficients are included.

Sample aliquots from this desorption test were analyzed twice. After running the samples in order, they were analyzed again in reverse order to quantify any effect of drift in the equipment and as a quality control check.

4.5 Test 2 – Concentration effects

Test 2 assessed the impact of concentration on K_d values. Surface soil from K7 was used in these tests. Tests were conducted following the methodology outlined in Section 3. Five different concentrations of NG and DNT at 0.10, 1, 10, 40, and 80 mg/L were to be evaluated according to the Work Plan (USACE 2007). This Work Plan originally specified concentrations of 50 and 100 mg/L. However, at the 100 mg/L concentration, a portion of the reagent-grade standard remained as a separate phase and would not completely dissolve into the water. Consequently, the maximum concentration level to be evaluated was lowered to 80 mg/L (Appendix B). The 50 mg/L test was also reduced to 40 mg/L to be consistent. Additionally, the results from Test 1, conducted at 24 hr with a concentration of 10 mg/L, were utilized for this series of tests.

Results indicate essentially no difference in NG adsorption K_d values with different solute concentrations (Table 9) determined from the individual experiments. In contrast, DNT adsorption K_d values decreased slightly with increasing concentration. There is greater variability in the desorption K_d values as compared to the adsorption K_d values, which is indicated by the higher standard deviations from the mean. In the DNT

Table 9. Adsorption and desorption partitioning coefficients (K_d) by spiked aqueous concentration for Tests 2 and 2d.

Test 2 - Evaluation of Adsorption K_d by Concentration					Test 2d - Evaluation of Desorption K_d by Concentration				
Conc. Sol*		K_d (L/kg)			Conc. Sol		K_d (L/kg)		
mg/L	Rep	2,4-DNT	2,6 DNT	NG	mg/L	Rep	2,4-DNT	2,6-DNT	NG
0.1	1	5.7	3.1	0.7	0.1	1	0.0	0.0	0.0
	1 dup**	6.0	4.1	0.3		NA*	NA	NA	NA
	2	5.5	3.2	0.1		2	5.7	1.3	BDL ††
	3	6.0	4.0	0.0		3	1.7	2.0	BDL
	1r	4.0	5.8	0.7		1r †	0.0	0.0	0.0
	1r dup	5.0	2.9	0.8		NA	NA	NA	NA
	2r	2.7	2.5	0.6		2r	4.6	0.0	BDL
	3r	4.2	3.1	1.1		3r	0.6	0.0	BDL
	Mean	4.9	3.6	0.5#		Mean	2.1#	0.6#	0.0#
1	1	4.9	3.6	1.0	1	1	6.6	4.2	0.0
	2	4.5	3.4	0.9		2	6.8	4.4	0.1
	3	4.7	3.5	0.9		3	5.7	3.2	0.0
	1r	5.0	3.9	0.8		1r	5.9	4.2	0.0
	2r	4.4	3.3	0.8		2r	6.4	4.1	0.0
	3r	4.8	3.3	0.5		3r	7.8	3.8	0.0
	Mean	4.7	3.5	0.8		Mean	6.5	4.0	0.02#
10	1	4.0	3.0	0.9	10	1	5.5	2.9	0.0
	2	4.0	2.9	1.0		2	5.4	3.0	0.0
	2 dup	NA	NA	NA		2 dup	5.4	3.0	0.0
	3	4.3	3.2	1.2		3	6.4	3.9	0.1
	1r	NA	NA	NA		1r	5.8	3.0	0.0
	2r	NA	NA	NA		2r	5.7	3.0	0.0
	2r dup	NA	NA	NA		2r dup	5.7	3.0	0.0
	3r	NA	NA	NA		3r	6.3	3.9	0.1
	Mean	4.1	3.0	1.0		Mean	5.8	3.2	0.03#
40	1	3.3	2.7	1.0	40	1	6.6	4.2	0.0
	2	3.2	2.6	1.0		2	6.8	4.4	0.1
	3	3.4	2.8	0.9		3	6.5	4.1	0.3
	1r	3.2	2.8	1.0		1r	4.9	5.5	0.3
	2r	3.2	2.8	1.0		2r	5.0	4.9	1.5
	3r	3.4	2.8	0.9		3r	5.3	5.0	1.3
	Mean	3.3	2.8	1.0		Mean	5.9	4.7	0.6#
80	1	2.8	2.4	0.8	80	1	3.1	3.8	0.0
	2	2.8	2.4	0.8		2	4.0	3.7	0.8
	3	2.8	2.3	0.9		3	4.1	3.7	0.8
	1r	2.8	2.4	0.8		1r	3.1	3.8	0.0
	2r	2.9	2.3	0.9		2r	4.1	3.7	0.8
	3r	2.9	2.3	0.9		3r	4.2	3.7	0.9
	Mean	2.8	2.3	0.9		Mean	3.8	3.7	0.6#
Overall Mean		4.0	3.1	0.8	Overall Mean		4.7#	2.9#	0.3#
Standard Deviation		1.0	0.7	0.3	Standard Deviation		2.0#	1.9#	0.4#
All tests used 14 g soil from K7 with 70 mL of DI at vary-					All tests used corresponding adsorption test soil with 70 mL of				

ing concentrations of NG and DNT.	DI added.
* Conc. Sol - solution concentrate, **NA – not applicable; *** dup – duplicate.	† - r reverse order of analysis; †† - BDL below detection limits.
# Calculated negative K_d values were assigned a value of zero for statistical calculations.	

desorption test the calculated K_d numbers are comparable to the adsorption K_d values. In contrast, several of the NG desorption K_d values are negative. This is an artifact of post-test aqueous concentration being slightly higher than the pre-test result. This may be due to NG not reaching equilibrium with the soil within 24 hr or simply due to experimental error in measuring small differences between small quantities. All negative values in Table 9 were arbitrarily set to zero to avoid the affect of unrealistic values on the means. In a number of desorption tests at the 0.1 mg/L concentration, no NG was detected with the HPLC. There was insufficient sample volume to reanalyze these samples with the GC-ECD, which has a lower detection limit.

Adsorption and desorption K_d can be readily determined if the data is linear with respect to concentration. Non-linear trends are usually consistent with Langmuir or Freundlich isotherm characteristics (Appendix C), which result in a concentration-dependant relationship between the solution and soil. The plots appear to be reasonably linear (Figures 8, 9, and 10) over the evaluated concentration range. The K_d values noted on the figures were determined using a least-squares fit regression analysis. The adsorption and desorption results for the spike concentration of 0.1 mg/L are probably erroneous in all three plots because of measurement uncertainty. Two of three test results yielded undetectable aqueous concentrations.

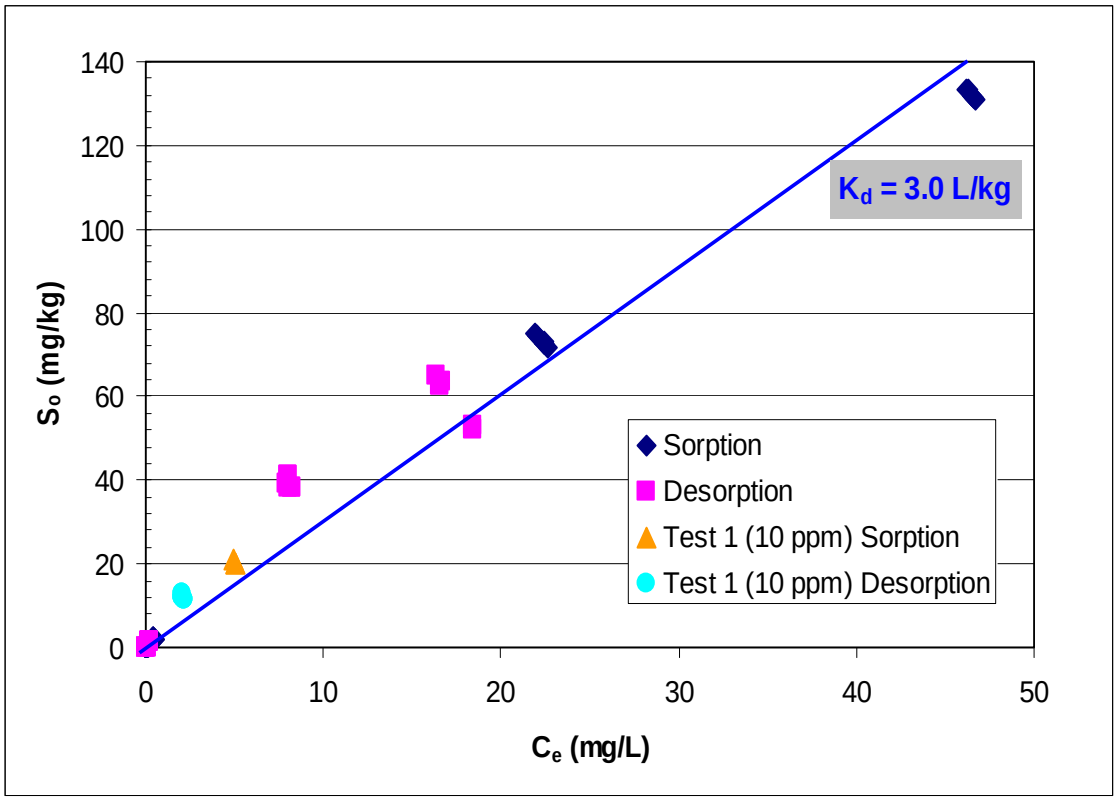

Figure 8. The 2,4-DNT concentration in the measured aqueous phase (C_e) versus the estimated soil concentration (S_o).

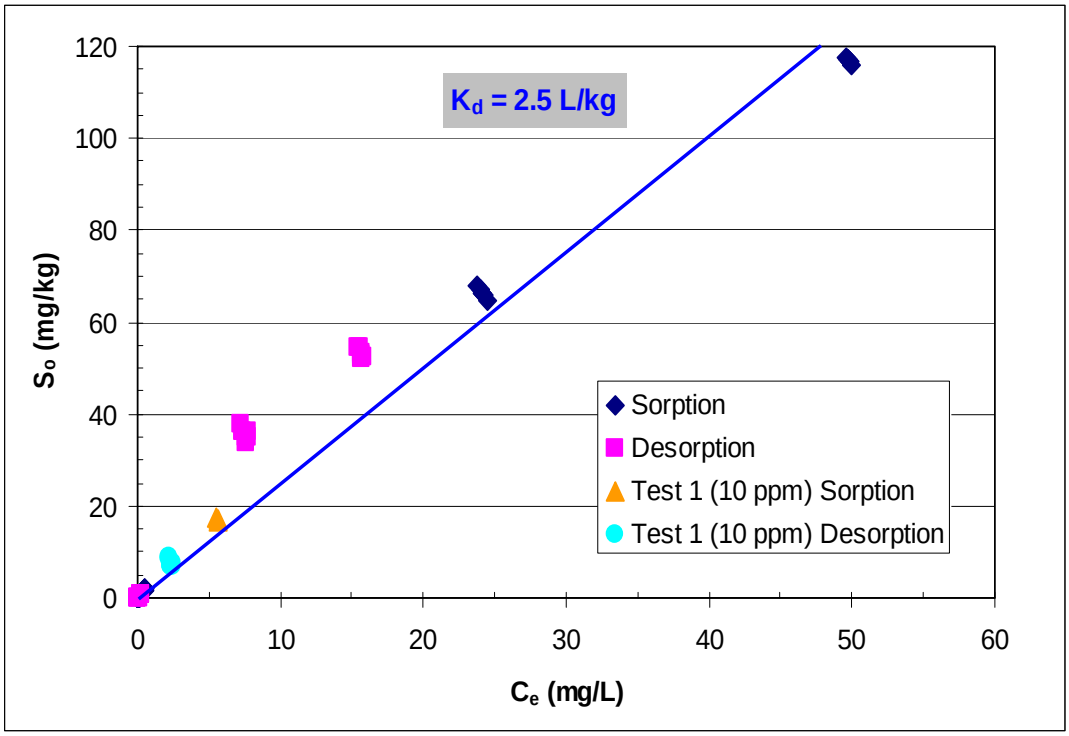

Figure 9. The 2,6-DNT concentration in the measured aqueous phase (C_e) versus the estimated soil concentration (S_o).

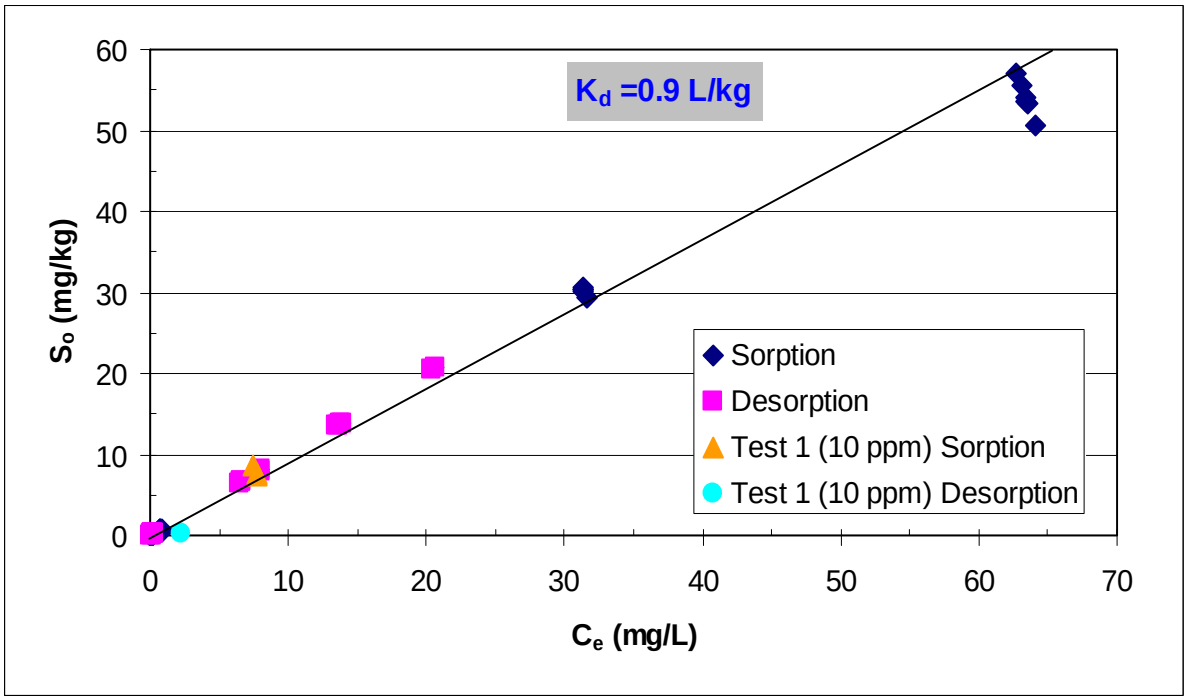

Figure 10. The NG concentration in the measured aqueous phase (C_e) versus the estimated soil concentration (S_o).

Two different approaches used to determine the mean K_ds included calculation of individual K_ds for each experiment as shown in Table 9 and performing linear regression on the data as shown in Figures 8 to 10. The mean adsorption and desorption K_ds over the concentration range studied are summarized in Table 10, and the mean results indicate that the different approaches yield slightly different K_d values.

Table 10. Summary of adsorption and desorption partitioning coefficients.

	Average K_d Table 9 (L/kg)		K_d Linear Regression, Figures 8 to 10 (L/kg)
	Adsorption	Desorption	
2,4 DNT	4.0	4.7	3.0
2,6 DNT	3.1	2.9	2.5
NG	0.8	0.3	0.9

The adsorption and the desorption DNT results suggest that a solution strength of 10 mg/L was a good middle ground for conducting the remainder of the tests (Tests 3 to 6). However, it is unclear from the data whether 10 mg/L was an appropriate concentration for the NG desorption tests. A higher concentration may have been more desirable from an analytical perspective. Although the soil samples collected for this study contained DNT and NG in the 0.1 to 70 mg/kg range (Table 1), the soil pore-water concentration in the field is unknown. As will be further discussed, insight regarding the pore-water concentrations cannot be derived from the column tests performed with fired propellant residue because no NG was observed in the effluent.

4.6 Test 3 - Heterogeneity

Test 3 evaluated the effect of heterogeneity on K_d values. Tests were conducted with two surface soil (0 - 3 in.) samples from each of the three ranges (Echo, Juliet, and Kilo). The specific soil samples utilized in this test were E1, E2, J1, J2, K6, and K7 (Table 11). The K1 and K2 samples were not tested as part of Test 3, due to the presence of a high concentration of NG. The adsorption test procedures used are outlined in Section 4.1

As discussed in Section 3.1, several samples utilized in the batch tests had low levels of NG and DNT present in the soil. Theoretically, the pre-existing mass of NG and DNT in these weathered soils should be accounted for when performing the K_d calculations. However, as discussed

in Section 4.11, the highly contaminated K1 and K2 soils did not leach NG and DNT into solution. It is assumed the lesser contaminated soils utilized in the batch Tests 3 and 4 from E, J, and K samples exhibited a similar degree of propellant weathering, and DNT and NG did not leach into solution. Therefore, there is no need for correction of the K_d to account for the pre-existing NG and DNT in soil for this test or Test 4.

Adsorption results indicate the K_d values for most soils are essentially the same (Table 11). The overall standard deviation for the adsorption K_d values ranged from 0.2 to 1.5 L/kg. It is possible the 2,4-DNT and 2,6-DNT adsorption K_d values are higher for the K7 soil than for other soils. However, too few tests were conducted to assess if this difference was statistically significant.

Desorption results are similar to adsorption results with most K_d values slightly higher than the adsorption values (Table 11). One exception is the NG desorption K_d value for the K7 soil, which was obtained as part of Test 1. These samples yielded a K_d value of zero. The K7-NG K_d values are largely responsible for the larger than desired standard deviation (Table 11). If the K7 soils are ignored in the calculation of the standard deviation, the value obtained is 0.9 L/kg.

Table 11. Surface soil heterogeneity evaluation of adsorption and desorption partitioning coefficients (K_d) for Tests 3 and 3d.

Test 3 - Evaluation of Adsorption K_d by Surface Soil Location					Test 3 - Evaluation of Desorption K_d by Surface Soil Location				
		K_d (L/kg)					K_d (L/kg)		
Location	Rep	2,4-DNT	2,6-DNT	NG	Location	Rep	2,4-DNT	2,6-DNT	NG
E1	1	2.1	1.9	0.7	E1	1	6.4	6.2	4.6
	1r	2.0	1.7	0.6		1r	5.9	5.5	4.1
	2	1.9	1.6	0.7		2	5.2	3.9	3.8
	2r	1.8	1.4	0.7		2r	5.0	3.1	3.9
	3	1.9	1.6	0.6		NA	NA	NA	NA
	3 dup	1.9	1.6	0.7		NA	NA	NA	NA
	3r	1.9	1.5	0.7		NA	NA	NA	NA
	3r dup	1.8	1.4	0.8		NA	NA	NA	NA
	Mean	1.9	1.6	0.7		Mean	5.6	4.7	4.1
E2	1	1.8	1.5	0.6	E2	1	5.7	5.5	3.1
	1r	1.8	1.4	0.5		1r	5.8	5.0	3.0
	2	1.7	1.5	0.6		2	4.0	2.5	1.8
	2r	1.7	1.4	0.6		2r	3.7	2.1	1.3
	3	1.9	1.5	0.6		NA	NA	NA	NA
	3r	2.0	1.5	0.8		NA	NA	NA	NA
	Mean	1.8	1.5	0.6		Mean	4.8	3.8	2.3
J1	1	2.1	1.7	0.6	J1	1	6.1	4.9	2.6
	1r*	2.0	1.8	0.5		1r	5.7	5.1	1.8
	2	2.2	1.8	0.6		2	5.0	3.3	1.9
	2r	2.3	1.8	0.9		2r	5.1	3.4	3.4
	2r	2.2	1.8	0.7		NA**	NA	NA	NA
	2r dup †	2.2	1.7	0.7		NA	NA	NA	NA
	3	2.3	1.9	0.7		NA	NA	NA	NA
	3r	2.4	1.8	0.6		NA	NA	NA	NA
	Mean	2.2	1.8	0.7		Mean	5.5	4.2	2.4
J2	1	3.2	2.6	1.0	J2	1	9.0	6.5	3.1
	1r	3.4	2.6	1.0		1r	9.2	6.5	2.9
	2	3.4	2.8	0.9		2	7.4	6.6	3.9
	2r	3.4	2.7	1.0		2r	7.4	6.4	4.0
	3	3.6	2.9	1.0		NA	NA	NA	NA
	3r	3.7	2.7	1.1		NA	NA	NA	NA
	Mean	3.5	2.7	1.0		Mean	8.2	6.5	3.5
K6	1	2.6	2.2	0.9	K6	1	6.2	5.1	2.3
	1r	2.7	2.1	0.8		1r	6.3	4.8	1.9
	2	2.8	2.4	1.0		2	5.4	4.3	2.7
	2r	2.9	2.1	0.8		2r	5.4	3.6	2.1
	3	2.9	2.5	0.9		NA	NA	NA	NA
	3r	3.0	2.3	0.8		NA	NA	NA	NA
	Mean	2.8	2.3	0.9		Mean	5.8	4.5	2.2

Table 11 (cont.). Surface soil heterogeneity evaluation of adsorption and desorption partitioning coefficients (K_d) for Tests 3 and 3d.

Test 3 - Evaluation of Adsorption K_d by Surface Soil Location					Test 3 - Evaluation of Desorption K_d by Surface Soil Location				
		K_d (L/kg)					K_d (L/kg)		
Location	Rep	2,4-DNT	2,6-DNT	NG	Location	Rep	2,4-DNT	2,6-DNT	NG
K7	1	4.0	3.0	0.9	K7	1	5.5	2.9	0.0
	2	4.0	2.9	1.0		2	5.4	3.0	0.0
	NA	NA	NA	NA		2 dup	5.4	3.0	0.0
	3	4.3	3.2	1.2		3	6.4	3.9	0.1
	NA	NA	NA	NA		1r	5.8	3.0	0.0
	NA	NA	NA	NA		2r	5.7	3.0	0.0
	NA	NA	NA	NA		2r dup	5.7	3.0	0.0
	NA	NA	NA	NA		3r	6.3	3.9	0.1
	Mean	4.1	3.0	1.0		Mean	5.8	3.2	0.03#
Overall Mean		2.5	2.0	0.8	Overall Mean		5.9	4.3	2.1#
Standard Deviation		0.8	0.6	0.2	Standard Deviation		1.1	1.3	1.5#
All tests used 14 g soil from varying locations with 70 mL of DI at concentration of 2,4-DNT = 9.32 ppm, 2,6-DNT = 9.35 mg/L, and NG= 9.28 mg/L					All tests used corresponding adsorption test soil with 70 mL of DI added. Note: Rep 3 samples sacrificed for soil analysis, except for K7.				
* r - reverse order of analysis; † dup - duplicate.					** NA – not applicable .				
# Calculated negative K_d values were assigned a value of zero for statistical calculations.									

4.7 Test 4 - Depth

Test 4 assessed the difference in K_d values by soil depth. The experiments were conducted with soil samples (E3 - E5, J3 - J5, and K3 - K5) collected from 9 to 12, 18 to 24, and 30 to 36 in. below ground surface (bgs) from each of the three ranges (Table 12). Data from Test 3 were utilized for comparison with the 0 to 3-in. interval. The adsorption and desorption methodology for the tests has been previously described in Section 4.1 and 4.2. The only variable changed in this test was the depth of the soil sample for each location.

As shown in Table 12, mean adsorption K_d values decreased with increasing soil depth for both DNTs and NG. Values were determined by calculating the mean using all data available for each depth interval (Tables 13, 14, and 15). The 2,4-DNT desorption K_d

Table 12. Summary of mean soil adsorption and desorption partitioning values (K_d) for 2,4-DNT, 2,6-DNT, and NG by depth for Tests 4 and 4d.

Depth	Adsorption Mean (L/kg)			Desorption Mean (L/kg)		
	(in. BGS)	2,6-DNT	NG	2,4-DNT	2,6-DNT	NG
0-3	2.3	1.8	0.7	5.6	4.4	2.9
9-12	1.1	0.8	0.2	5.7	7.8	3.7

18-24	0.7	0.4	0.1	5.1	9.3	2.4
30-36	0.5	0.3	0.1	5.2	6.6	1.6

values appear to decline slightly with increasing depth (Fig. 11). However, desorption K_ds for 2,6-DNT do not appear to decline with depth. NG desorption K_d values appear to decline with depth, but there are too few sample intervals to conclusively make this determination. The desorption K_d values were consistently higher than the adsorption K_ds. This was true for the soils at E, J, and K Ranges (Tables 13, 14, and 15). Speitel et al. (2002) attributed a decrease in adsorption K_ds with increasing depth to a decline in OC content. The results in Table 12 suggest that the decline in adsorption K_d with increasing depth may also be related to declining OC content, or possibly a decline in the CEC. Figures 11 and 12 are plotted using samples that had OC, CEC and K_d measurements and used the mean K_d value for all samples for a given depth interval. As CEC decreases with increasing soil depth so do the K_ds for NG and DNT. Yamamoto et al. (2004) discuss the irreversibility (Figure 12) between adsorption and desorption (as indicated by a higher desorption K_d value) for DNTs. However, it was not determined if weathering of contaminated soils reduced availability of the contaminant for desorption, and hence resulted in a higher K_d than for adsorption. Results of tests with both spiked and weathered soils (see the discussion of results of subsequent tests with weathered soils) clearly demonstrates weathering results in the loss of any NG available on the surface of the NC (Table 8d). Any remaining NG is encapsulated within the NC matrix, which limits its release. This dissolution process is described more fully in Appendix A.

Without further study, it is not possible to identify whether CEC, OC, or both, are responsible for a decline in the K_d values with increasing soil depth. A possible regression equation could be developed so that TOC and CEC soil measurements could be used to predict the 2,4-DNT and NG K_d values without the need for performing batch experiments.

Table 13. Effect of soil depth on adsorption and desorption partitioning coefficients (K_d) for Kilo Range soils for Tests 4 and 4d.

Test 4 - Evaluation of Adsorption K_d by Depth for Kilo Range Soils						Test 4d - Evaluation of Desorption K_d by Depth for Kilo Range Soils					
	Depth		K_d (L/kg)				Depth		K_d (L/kg)		
Location	(in. bgs)	Rep	2,4-DNT	2,6-DNT	NG	Location	(in. bgs)	Rep	2,4-DNT	2,6-DNT	NG
K6	0-3	1	2.6	2.2	0.9	K6	0-3	1	6.2	5.1	2.3
		1r*	2.7	2.1	0.8			1r	6.3	4.8	1.9
		2	2.8	2.4	1.0			2	5.4	4.3	2.7
		2r	2.9	2.1	0.8			2r	5.4	3.6	2.1
		3	2.9	2.5	0.9			NA**	NA	NA	NA
		3r	3.0	2.3	0.8			NA	NA	NA	NA
		Mean	2.8	2.3	0.9			**Mean**	5.8	4.5	2.2
K3	9 - 12	1	1.3	0.9	0.5	K3	9 - 12	1	5.7	0.0	0.0
		1r	1.3	1.0	0.4			1r	5.4	0.4	0.0
		2	1.3	0.8	0.3			2	5.8	0.1	0.0
		2r	1.3	0.8	0.3			2r	5.9	0.2	0.0
		3	1.3	0.9	0.3			3	5.1	0.0	0.0
		3r	1.2	0.8	0.4			3r	4.5	-0.2	0.0
		Mean	1.3	0.9	0.4			**Mean**	5.4	0.1	0.0#
K4	18 - 24	1	0.9	0.6	0.3	K4	18 - 24	1	4.1	0.0	0.0
		1r	0.9	0.5	0.3			1r	4.0	0.0	0.0
		1 dup †	0.9	0.5	0.2			NA	NA	NA	NA
		1r dup	0.8	0.5	0.2			NA	NA	NA	NA
		2	0.8	0.4	0.2			2	5.6	0.0	0.0
		2r	0.8	0.4	0.1			2r	5.5	0.0	0.0
		3	1.0	0.5	0.2			3	6.5	0.0	0.0
		3r	1.0	0.5	0.3			3r	6.5	0.0	0.0
		Mean	0.9	0.5	0.2			**Mean**	5.4	0.0#	0.0#
K5	30 - 36	1	0.7	0.4	0.3	K5	30 - 36	1	7.4	0.0	0.0
		1r	0.7	0.4	0.2			1r	7.5	0.0	0.0
		2	0.7	0.3	0.3			2	7.6	0.0	0.0
		2r	0.7	0.4	0.2			2r	6.5	0.0	0.0
		3	0.6	0.3	0.2			3	5.9	0.0	0.0
		3r	0.6	0.2	0.2			3r	5.5	0.0	0.0
		Mean	0.7	0.3	0.2			**Mean**	6.7	0.0#	0.0#
All tests used 14 g soil from varying locations with 70 mL of DI at concentration of 2,4-DNT = 9.32 mg/L,, 2,6-DNT = 9.35 mg/L, and NG= 9.28 mg/L .						All tests used corresponding adsorption test soil with 70 mL of DI added.					
* r - reverse order of analysis; † dup - duplicate.						** NA – not applicable.					
# Calculated negative K_d values were assigned a value of zero for statistical calculations.											

Table 14. The effect of soil depth on the adsorption and desorption partitioning coefficients (k_d) for Echo Range soils for Tests 4 and 4d.

Test 4 - Evaluation of Adsorption K_d by Depth for Echo Range Soils						Test 4d - Evaluation of Desorption K_d by Depth for Echo Range Soils					
	Depth		K_d (L/kg)				Depth		K_d (L/kg)		
Location	(in. bgs)	Rep	2,4-DNT	2,6-DNT	NG	Location	(in. bgs)	Rep	2,4-DNT	2,6-DNT	NG
E1	0-3	1	2.1	1.9	0.7	E1	0-3	1	6.4	6.2	4.6
		1r*	2.0	1.7	0.6			1r	5.9	5.5	4.1
		2	1.9	1.6	0.7			2	5.2	3.9	3.8
		2r	1.8	1.4	0.7			2r	5.0	3.1	3.9
		3	1.9	1.6	0.6			NA**	NA	NA	NA
		3r	1.9	1.6	0.7			NA	NA	NA	NA
		3 dup †	1.9	1.5	0.7			NA	NA	NA	NA
		3r dup	1.8	1.4	0.8			NA	NA	NA	NA
		Mean	**1.9**	**1.6**	**0.7**			**Mean**	**5.6**	**4.7**	**4.1**
E3	9 - 12	1	1.3	1.0	0.2	E3	9 - 12	1	6.8	11.1	6.7
		1r	1.3	1.0	0.2			1r	7.0	11.6	6.2
		2	1.2	0.8	0.1			2	6.6	10.7	5.2
		2r	1.2	0.8	0.4			2r	6.6	10.3	8.7
		3	1.3	0.9	0.2			3	6.8	10.9	6.7
		3r	1.2	0.8	0.2			3r	6.7	10.8	6.7
		Mean	**1.3**	**0.9**	**0.2**			**Mean**	**6.8**	**10.9**	**6.7**
E4	18 - 24	1	0.6	0.6	0.3	E4	18 - 24	1	5.4	13.4	10.1
		1r	0.6	0.5	0.3			1r	5.4	12.8	9.0
		2	0.5	0.5	-0.2			2	4.7	12.4	0.0
		2r	0.6	0.5	0.1			2r	5.3	12.5	5.8
		3	0.5	0.5	0.1			3	4.3	11.9	5.6
		3r	0.6	0.4	0.2			3r	4.6	10.6	9.1
		Mean	**0.6**	**0.5**	**0.1**			**Mean**	**4.9**	**12.3**	**6.6**
E5	30 - 36	1	0.7	0.5	0.0	E5	30 - 36	1	5.7	11.7	3.1
		1r	0.8	0.5	0.2			1r	6.8	11.6	8.1
		2	0.9	0.7	0.2			2	7.4	14.0	7.1
		2r	0.9	0.6	0.2			2r	7.7	12.9	8.5
		3	0.9	0.7	0.0			3	6.8	12.2	3.8
		3r	1.1	0.8	0.1			3r	7.8	13.5	5.6
		Mean	**0.9**	**0.6**	**0.1**			**Mean**	**7.0**	**12.6**	**6.0**
All tests used 14 g soil from varying locations with 70 mL of DI at concentration of 2,4-DNT = 9.32 mg/L, 2,6-DNT = 9.35 mg/L,, and NG= 9.28 mg/L.						All tests used corresponding adsorption test soil with 70 mL of DI added.					
* r - reverse order of analysis; † dup - duplicate.						** NA – not applicable.					
# Calculated negative K_d values were assigned a value of zero for statistical calculations.											

Table 15. Effect of soil depth on the adsorption and desorption partitioning coefficients (K_d) for Juliet Range soils for Tests 4 and 4d.

Test 4 - Evaluation of Adsorption K_d by Depth for Juliet Range Soils						Test 4d - Evaluation of Desorption K_d by Depth for Juliet Range Soils					
	Depth		K_d (L/kg)				Depth		K_d (L/kg)		
Location	(in. bgs)	Rep	2,4-DNT	2,6-DNT	NG	Location	(in. bgs)	Rep	2,4-DNT	2,6-DNT	NG
J1	0-3	1	2.1	1.7	0.6	J1	0-3	1	6.1	4.9	2.6
		1r*	2.0	1.8	0.5			1r	5.7	5.1	1.8
		2	2.2	1.8	0.6			2	5.0	3.3	1.9
		2r	2.3	1.8	0.9			2r	5.1	3.4	3.4
		2 dup †	2.2	1.8	0.7			NA**	NA	NA	NA
		2r dup	2.2	1.7	0.7			NA	NA	NA	NA
		3	2.3	1.9	0.7			NA	NA	NA	NA
		3r	2.4	1.8	0.6			NA	NA	NA	NA
		Mean	**2.2**	**1.8**	**0.7**			**Mean**	**5.5**	**4.2**	**2.4**
J3	9 - 12	1	0.7	0.6	0.0	J3	9 - 12	1	4.7	12.3	3.5
		1r	0.8	0.4	0.3			1r	5.2	10.2	7.6
		2	0.8	0.7	0.1			2	5.1	12.3	4.9
		2r	0.7	0.5	0.2			2r	4.4	10.9	6.6
		3	0.9	0.7	0.1			3	5.2	12.5	5.0
		3r	1.0	0.7	0.2			3r	5.6	12.1	6.5
		Mean	**0.8**	**0.6**	**0.2**			**Mean**	**5.0**	**11.7**	**5.7**
J4	18 - 24	1	0.7	0.2	0.0	J4	18 - 24	1	5.3	16.3	4.6
		1r	0.6	0.2	0.0			1r	4.8	17.5	2.1
		2	0.6	0.3	0.0			2	4.9	19.0	4.0
		2r	0.6	0.2	0.0			2r	5.2	18.0	4.1
		3	0.6	0.2	0.0			3	4.9	16.1	2.4
		3r	0.6	0.2	0.0			3r	5.0	16.7	4.8
		Mean	**0.6**	**0.2**	**0.0#**			**Mean**	**5.0**	**17.3**	**3.7**
J5	30 - 36	1	0.2	0.2	0.1	J5	30 - 36	1	5.3	28.0	6.3
		1r	0.2	0.1	0.1			1r	5.3	27.3	5.8
		2	0.0	0.0	0.0			2	0.0	0.0	0.0
		2r	0.0	0.0	0.0			2r	0.0	0.0	0.0
		2r dup	0.0	0.0	0.0			2r dup	NA	NA	NA
		3	0.0	0.0	0.0			3	0.0	0.0	0.0
		3r	0.0	0.0	0.0			3r	0.0	0.0	0.0
		Mean	**0.0**	**0.0**	**0.0**			**Mean**	**1.8**	**9.2**	**2.0**
All tests used 14 g soil from varying locations with 70 mL of DI at concentration of 2,4-DNT = 9.32 mg/L, 2,6-DNT = 9.35 mg/L,, and NG= 9.28 mg/L.						All tests used corresponding adsorption test soil with 70 mL of DI added.					
* r - reverse order of analysis; † dup - duplicate.						** NA – not applicable.					
# Calculated negative K_d values were assigned a value of zero for statistical calculations.											

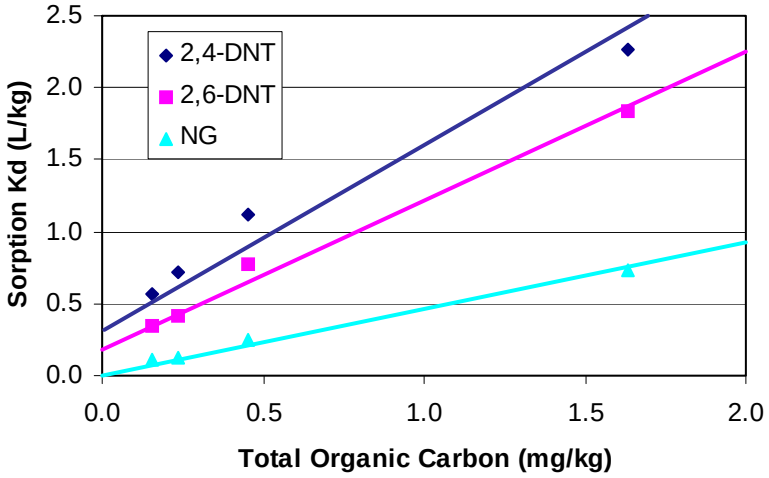

Figure 11. Adsorption K_d versus Total OC.

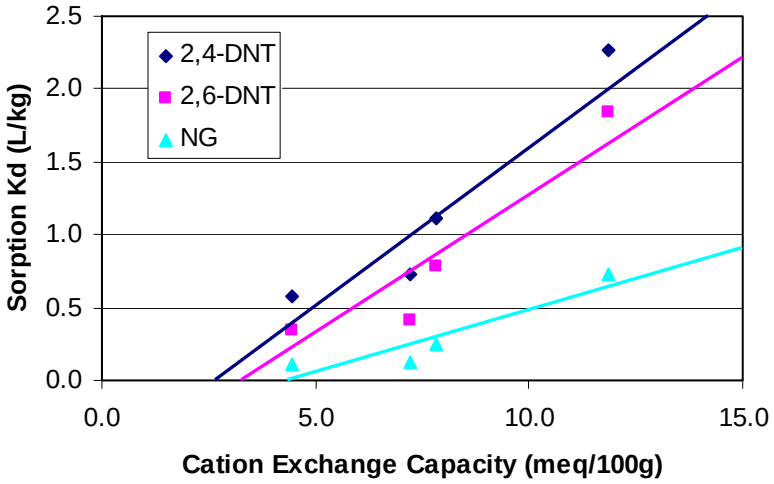

Figure 12. Adsorption K_d versus CEC.

4.8 Test 5 – Temperature

In Test 5, the high temperature reaction was equilibrated at 32°C, and the low temperature reaction was equilibrated at 12°C. Room temperature was 25°C with results obtained from the Test 1 experiments conducted at 24 hr. Test 5 utilized the K7 soil and the methodology for the adsorption and desorption experiments described in Section 4.1 and 4.2. Test 1 results were conducted at a spiked concentration of approximately 10 mg/L for 24 hr and were used for the mid temperature comparison. Although a steady state was demonstrated in Test 1, equilibrium may not have been established for desorption. As discussed in Section 4.1, some negative values for

desorption coefficients were calculated but were arbitrarily set at zero for calculation purposes.

No significant difference in the NG adsorption or desorption K_d values was observed over the temperature range studied (Table 16). However, K_d for DNTs does appear to decline somewhat with increasing temperature. As observed for other tests. DNT desorption K_d values were typically higher than the adsorption K_d values although NG values were essentially zero.

Table 16. Evaluation of partitioning coefficients (K_d) with temperature.

Test 5 - Evaluation of Adsorption K_d by Temperature					Test 5d - Evaluation of Desorption K_d by Temperature				
Temp		K_d (L/kg)			Temp		K_d (L/kg)		
°C	Replicate	2,4-DNT	2,6-DNT	NG	°C	Replicate	2,4-DNT	2,6-DNT	NG
12	1	4.1	3.1	0.9	12	1	7.4	6.9	0.0
	1 dup*	4.2	3.2	0.9		NA**	NA	NA	NA
	2	5.1	3.8	1.0		2	7.5	7.1	0.0
	3	4.9	3.6	1.0		3	7.3	6.8	0.0
	NA	NA	NA	NA		1r †	7.2	6.2	0.0
	NA	NA	NA	NA		2r	7.5	6.5	0.0
	NA	NA	NA	NA		3r	7.3	6.5	0.0
	Avg	**4.6**	**3.4**	**1.0**		**Avg**	**7.4**	**6.7**	0.0#
25	1	4.0	3.0	0.9	25	1	5.5	2.9	0.0
	2	4.0	2.9	1.0		2	5.4	3.0	0.0
	2 dup	NA	NA	NA		2 dup	5.4	3.0	0.0
	3	4.3	3.2	1.2		3	6.4	3.9	0.1
	1r †	NA	NA	NA		1r	5.8	3.0	0.0
	2r	NA	NA	NA		2r	5.7	3.0	0.0
	2r dup	NA	NA	NA		2r dup	5.7	3.0	0.0
	3r	NA	NA	NA		3r	6.3	3.9	0.1#
	Avg	**4.1**	**3.0**	**1.0**		**Avg**	**5.8**	**3.2**	**0.03**
32	1	3.3	2.5	0.9	32	1	3.5	3.2	0.0
	2	3.2	2.3	0.9		2	4.9	4.4	0.0
	3	3.5	2.5	1.0		3	4.3	3.4	0.0
	NA	NA	NA	NA		1r	3.5	2.8	0.0
	NA	NA	NA	NA		2r	4.9	4.4	0.0
	NA	NA	NA	NA		3r	4.2	3.2	0.0
	Avg	**3.3**	**2.4**	**0.9**		**Avg**	**4.2**	**3.6**	0.0#
All tests used 14 g soil from K7 with 70 mL DI at a concentration of 8.938 mg/L for 2,4-DNT, 8.89 mg/L NG, and 8.288 mg/L for 2,6-DNT.					All tests used corresponding adsorption test soil with 70 mL of DI added.				
* duplicate; † r - reverse order of analysis.					** NA – not applicable.				
# Calculated negative K_d values were assigned a value of zero for statistical calculations.									

Temperature appears to have a slight effect on DNT K_d with values declining with increasing temperature. Differences were not evaluated statistically due to the lack of adequate data sets. It is not clear what mechanism is responsible for this difference as temperature had no effect on NG K_d values. The results suggest that if DNT is released to the environment in an aqueous phase such as during a precipitation event, it might adsorb to soil less readily during periods of elevated temperatures. This effect is expected to be slight, and temperature variations tend to dampen quickly with depth.

4.9 Test 6 - pH

In Test 6, the effect of soil pH on the K_d of NG and DNT was investigated. Surface soil (0 to 3 in.) from K7 was used in this experiment following the procedures described in Section 4.1 and 4.2. The median soil pH of three studied SARs was 6.4, which compares to a pH of 7.3 for the K7 soil. A YSI meter, model 556 MPS, was used to measure soil pH. Soil pH was obtained by mixing one part DI with one part soil. Effects of changes in soil pH on NG and DNT adsorption were evaluated by adjusting the initial pH of the K7 soil to approximately pH 4.6 and 8.2, respectively.

The Work Plan (USACE 2007) indicated that hydrochloric acid would be used to lower soil pH (Appendix B). However, after consultation with several CRREL soil scientists, it was decided to use a weaker acid. Concern was expressed that a strong acid such as hydrochloric acid might dissolve part of the soil matrix, and change soil properties. Therefore, to lower soil pH, 29.25 mL of 0.2 M boric acid and 0.75 mL of 0.05 M citric acid were mixed together using a process outlined in Shugar and Ballinger (1990). A solution of 0.1 M tertiary sodium phosphate was added to the first solution mixture and then added to 14 g of soil. The soil pH was thus lowered to 4.2.

In addition, the Work Plan (USACE 2007) called for the use of sodium hydroxide to raise the soil pH (Appendix B). However, concern was expressed that sodium hydroxide might potentially initiate hydrolysis of the NG and DNT. Therefore, to raise soil pH, boric acid, citric acid, and tertiary sodium phosphate were initially used in a different proportion (Shugar and Ballinger 1990). However, this method did not satisfactorily raise pH to the desired level. The addition of sodium phosphate proved successful in raising soil pH to an average of 8.2. Approximately 7.5 g sodium phosphate mixed with 70 mL DI was added to 14 g of soil.

Results indicate little effect of pH on the soil and K_d values (Table 17), raising the possibility that materials used to modify the soil pH are responsible for the inconsistent results between low and high pH.

Table 17. Evaluation of partitioning coefficients (K_d) by soil pH.

Test 6 - Evaluation of Adsorption K_d by pH					Test 6d - Evaluation of Desorption K_d by pH				
		K_d (L/kg)					K_d (L/kg)		
pH	**Replicate**	**2,4-DNT**	**2,6-DNT**	**NG**	**pH**	**Replicate**	**2,4-DNT**	**2,6-DNT**	**NG**
Low	1	5.5	6.8	2.9	Low	1	5.5	10.0	0.5
(4.2)	1 dup*	5.6	6.9	3.1	(4.2)	1 dup	NA**	NA	NA
	1r	5.4	6.9	3.1		1r	5.6	10.2	0.9
	1r dup	5.8	7.1	3.3		1r dup	NA	NA	NA
	2	5.5	7.0	3.1		2	5.3	9.6	0.5
	2r †	5.5	7.0	3.2		2r	5.4	9.7	0.7
	3	5.5	6.9	3.1		3	5.7	9.7	1.0
	3r	5.9	7.1	3.0		3r	5.9	10.2	1.1
	Mean	**5.6**	**7.0**	**3.1**		**Mean**	**5.6**	**9.9**	**0.8**
Neutral	1	4.0	3.0	0.9	Neutral	1	5.5	2.9	0.0
(7.3)	2	4.0	2.9	1.0	(7.3)	2	5.4	3.0	0.0
	2 dup	NA	NA	NA		2 dup	5.4	3.0	0.0
	3	4.3	3.2	1.2		3	6.4	3.9	0.1
	1r	NA	NA	NA		1r	5.8	3.0	0.0
	2r	NA	NA	NA		2r	5.7	3.0	0.0
	2r dup	NA	NA	NA		2r dup	5.7	3.0	0.0
	3r	NA	NA	NA		3r	6.3	3.9	0.1
	Mean	**4.0**	**3.0**	**1.0**		**Mean**	**5.8**	**3.2**	**0.03#**
High	1	8.7	5.9	2.2	High	1	5.8	5.6	0.9
(8.2)	2	7.8	5.6	2.0	(8.2)	2	5.4	5.5	0.8
	3	8.1	6.5	2.1		3	5.3	5.9	0.8
	1r	NA	NA	NA		1r	6.0	6.0	1.2
	2r	NA	NA	NA		2r	5.7	5.7	1.1
	3r	NA	NA	NA		3r	5.4	5.8	1.3
	Mean	**8.2**	**6.0**	**2.1**		**Mean**	**5.6**	**5.8**	**1.0**
All tests used 14 g soil from K7 with 70 mL DI at a concentration of 8.535 mg/L for 2,4-DNT, 9.105 mg/L NG, and 9.165 mg/L for 2,6-DNT.					All tests used corresponding adsorption test soil with 70 mL of DI added.				
* dup - duplicate; † r - reverse order of analysis.					** NA – not applicable.				
# Calculated negative K_d values were assigned a value of zero for statistical calculations.									

4.10 Test 7d – Unfired propellant

Although not specifically mandated in the Work Plan (USACE 2007), Test 7 was carried out to assess the potential difference in partitioning coefficients by adding unfired propellant to the K7 soil (Appendix B). In this test, 0.14 g of unfired solid propellant was added to 14 g of soil with no biocide. Unfired propellant was derived from an M855 projectile (5.56 mm) containing the propellant WC844. WC844 propellant typically is a mixture containing 66.95% NC, 11.2% NG, and 21.75% additives (MIDAS 2008). Individual propellant grains contain a graphite coating. Thus, based on MIDAS (2008), the estimated equivalent soil concentration was 1,120 mg/kg NG, which was comparatively high when compared to the values in Table 1. From a different perspective, if all the NG dissolved, it would yield a concentration of 224 mg/L (for example, 11.2% of 0.14 g NG in 70 mL of DI). No DNT exists in this propellant according to MIDAS (2008). Additionally, a sample of 0.14 g of unfired propellant was added to 70 mL DI and placed on the shaker table for 24 hr. Analysis revealed an aqueous concentration of 1.1 mg/L NG, indicating that less than 1% of the total mass of NG present in the unfired propellant had dissolved into solution over 24 hr.

In this test, the unfired propellant/soil mix was initially subjected to dissolution upon contact with water with and without the addition of biocide (Table 18). As no DNT was present in the propellant; the absence of DNT in the test results is consistent with the known propellant properties. It is clear only a small portion of the NG, 9 mg/L average (4% of

Table 18. Soil (desorption) partitioning coefficients (K_d) for unfired propellant.

Test 7d - Evaluation of Desorption of Unfired WC844 Propellant without Biocide				Test 7d - Evaluation of Desorption of Unfired WC844 Propellant with Biocide			
	K_d (L/kg)				K_d (L/kg)		
Replicate	2,4-DNT	2,6-DNT	NG	Replicate	2,4-DNT	2,6-DNT	NG
1	ND*	ND	0.9	1	ND	ND	10.3
2	ND	ND	1.2	2	ND	ND	10.5
3	ND	ND	0.6	3	ND	ND	7.9
1r †	ND	ND	0.9	1r	NA**	NA	NA
2r	ND	ND	1.1	2r	NA	NA	NA
3r	ND	ND	0.6	3r	NA	NA	NA
Mean	**ND**	**ND**	**0.9**	**Mean**	**ND**	**ND**	**9.6**
Test consisted of using 14 g K7 soil with 0.14 g of propellant with no biocide added. The WC844 propellant contains 11.2% NG and no DNT. This yields a soil concentration				Test consisted of using 14 g K7 soil with 0.14 g of propellant with biocide added. The WC844 propellant contains 11.2% NG and no DNT. This yields a soil concentration of 1120			

of 1120 51 mg/kg. Then 70 mL of DI is added.	51 mg/kg. Then 70 mL of DI is added.
* ND – not detected; **NA – not analyzed;† r - reverse order of analysis.	

the original total), was measured in the aqueous phase of the batch test with biocide. Therefore, release of the NG is likely being limited by the NC matrix because these two nitro esters have a high affinity to remain chemically bonded together. The experiment conducted without biocide yielded approximately 0.8 mg/L of NG in solution, suggesting biodegradation may be responsible for the additional loss. The difference might also be attributed to experimental error but was not evaluated due to the number of tests necessary.

4.11 Test 8d –Contaminated (weathered) soil

Test 8 involved desorption of NG and DNT from weathered K1 and K2 soils contaminated with fired propellant. K1 soil had a NG soil concentration of 79 mg/kg and 2,4-DNT concentration of 1.5 mg/kg (Table 1). K2 soil had an NG concentration of 42 mg/kg and a 2,4-DNT concentration of 1.1 mg/kg. No 2,6-DNT was detected in either soil sample. As in all the batch desorption tests, 70 mL of DI was added to 14 g of soil. Two tests were conducted — with and without adding biocide (Table 19).

Table 19. Evaluation of adsorption/desorption/dissolution for contaminated soil with and without biocide added.

Test 8 - Evaluation of Dissolution/Desorption from Contaminated Soil without Biocide					Test 8d - Evaluation of Dissolution/ Desorption from Contaminated Soil with Biocide				
		Concentration (mg/L)					Concentration (mg/L)		
Soil	Rep	2,4-DNT	2,6-DNT	NG	Soil	Rep	2,4-DNT	2,6-DNT	NG
K1	1	ND*	ND	ND	K1	1	ND	ND	ND
	1r	ND	ND	ND		1r	ND	ND	ND
	2	ND	ND	ND		2	ND	ND	ND
	2r**	ND	ND	ND		2r	ND	ND	ND
	3	ND	ND	ND		3	ND	ND	ND
	3r	ND	ND	ND		3r	ND	ND	ND
	Avg	**ND**	**ND**	**ND**		**Avg**	**ND**	**ND**	**ND**
K2	1	ND	ND	ND	K2	1	ND	ND	ND
	1r	ND	ND	ND		1r	ND	ND	ND
	2	ND	ND	ND		2	ND	ND	ND
	2r	ND	ND	ND		2r	ND	ND	ND
	3	ND	ND	ND		3	ND	ND	ND
	3r	ND	ND	ND		3r	ND	ND	ND

	Avg	ND	ND	ND		Avg	ND	ND	ND
Test consisted of using 14 g K1 or K2 soil and 70 mL of DI added and no biocide. The K1 soil had initial 2,4-DNT concentration of 1.51 mg/kg, 2,6-DNT = < 0.018 51 mg/kg, and NG = 69.64 51 mg/kg. The K2 soil had an initial 2,4-DNT concentration of 1.14 51 mg/kg, 2,6-DNT = < 0.018 51 mg/kg, and NG = 41.50 51 mg/kg.					Test consisted of using 14 g K1 or K2 soil and 70 mL of DI and biocide added. The K1 soil had initial 2,4-DNT concentration of 1.51 51 mg/kg, 2,6-DNT = < 0.018 51 mg/kg, and NG = 69.64 mg/kg. The K2 soil had an initial 2,4-DNT concentration of 1.14 51 mg/kg, 2,6-DNT = < 0.018 51 mg/kg, and NG = 41.50 51 mg/kg.				
*ND – Not detected. See MDL listing in Table 3. ** r – reverse order of analysis.									

No NG or DNT was measured in the aqueous phase after 24 hr of contact (Table 19) either with or without biocide. These results suggest desorption of residual, encapsulated NG and DNT present in the soils in the NC matrix, prior to the adsorption experiments used in the proceeding tests, does not need to be taken into account.

4.12 Test 9 – Rain water

A ninth test assessed any differences due to different ionic strength and pH of natural precipitation as compared to the use of DI. Precipitation collected by CENAE in a stainless steel bowl from Camp Edwards on April 27, 2007 was used in this series of tests. After collection, rainwater was kept in plastic bottles in the dark in a refrigerator at 4°C at CRREL. The general experimental procedures as outlined in Test 3 were followed for this test (for example, 24-hr duration, approximately 10 mg/L spiking concentration, and at room temperature). In addition to using biocide, the same soil without the addition of a biocide (K7) was also evaluated.

Surprisingly, the adsorption K_ds with biocide added to rain water were five to six times higher than those with DI and biocide for all three compounds (data not shown). Also, surprising was that adsorption K_ds were generally higher than the desorption K_ds. This is contrary to Tests 1 through 6 with DI where the desorption K_ds were always higher than adsorption K_ds.

Because of unusual results with old rainwater, a fresh rainwater sample was collected on May 5, 2008, and the tests were repeated. Upon receipt at CRREL, the measured rainwater sample properties were: pH of 4.7; specific conductance of 63 μS/cm; redox 250 millivolts; and a total dissolved solids reading of 0.041 g/L. Results of the fresh rainwater with biocide tests were similar to those obtained with DI and biocide for all three ana-

lytes (Table 20). The rainwater samples without biocide had slightly higher adsorption K_d values, presumably due to biodegradation processes.

Desorption K_d numbers for DI and rainwater biocide experiments are slightly higher than adsorption values, which is consistent with earlier tests. However, very small amounts of three compounds were present in the aqueous phase at the end of the desorption experiment with rainwater and no biocide, resulting in a very high apparent K_d value. Although reported values for rainwater without biocide were not true K_d values, for illustrative purposes it is useful to present results for this experiment in this manner. The high apparent K_d values indicate active biodegradation of all three

Table 20. Comparison of adsorption and desorption apparent K_d values for soil using DI and rain water from Camp Edwards.

Test 9 - Evaluation of Adsorption apparent K_d with Different Water					Test 9d - Evaluation of Desorption apparent K_d with Different Water				
		K_d (L/kg)					K_d (L/kg)		
Type	Rep	2,4-DNT	2,6-DNT	NG	Type	Rep	2,4-DNT	2,6-DNT	NG
DI	1	4.0	2.9	0.9	DI	1	9.9	7.5	2.6
	2	4.0	2.9	0.9		2	9.8	7.5	2.8
	NA*	NA	NA	NA		2 dup**	9.8	7.3	2.6
	3	4.3	3.1	1.1		3	10.8	8.5	3.7
	NA	NA	NA	NA		1r †	10.2	7.5	2.6
	NA	NA	NA	NA		2r	10.1	7.6	2.8
	NA	NA	NA	NA		2r dup	10.1	7.6	2.8
	NA	NA	NA	NA		3r	10.4	8.2	3.5
	Avg	**4.1**	**3.0**	**1.0**		**Avg**	**10.1**	**7.7**	**2.9**
Rainwater	1	0.0	2.7	0.8	Rainwater	1	9.7	11.6	3.5
w/Biocide	2	3.9	3.1	0.8	w/Biocide	2	11.6	15.4	4.3
5/5/2008	3	3.9	3.1	0.9	5/5/2008	3	10.8	13.7	5.0
	Avg	**2.6**	**3.0**	**0.9**		**Avg**	**10.7**	**13.5**	**4.3**
Rainwater	1	8.8	3.9	2.8	Rainwater	1	1052	209	BDL
without	2	6.5	3.3	1.9	without	2	BDL	BDL	619
Biocide	3	6.6	3.5	2.2	Biocide	3	239	54	BDL
5/5/2008	**Avg**	**7.3**	**3.6**	**2.3**	5/5/2008	**Avg**	**645**	**131**	**619**
* NA – not analyzed; **dup – duplicate; † r – reverse order of analysis.									

compounds, and the biodegradation component in relative terms is significantly more important than adsorption/desorption.

Tests conducted with rainwater and biocide resulted in results similar to those obtained with DI and biocide. However, greater differences were evident in experiments conducted without biocide. The rainwater samples had much less NG and DNT in the aqueous phase, suggesting that microorganisms facilitated the biological reduction and/or inorganic interactions with minerals or ions.

4.13 Test 10d – Fresh-fired propellant

Test 10d was conducted in the same manner as Test 7d. Namely, 0.14 g of freshly-fired propellant (a half-and-half mixture of 9 and 5.56 mm of propellant), obtained as described in Section 3.2.2, was added to 14 g of K7 soil. Seventy mL of DI was added either with biocide or without biocide, and the experiment was conducted at room temperature. At the end of this test, the NG concentration was measured in soil samples for each experiment, yielding an average concentration of 1,890 mg/kg for the three samples with biocide. Because the soil mass used in the experiments was small, the sample could not be ground. In addition, the entire sample (14 g) was not extracted with acetonitrile; instead 10 g was extracted, consistent with EPA Method 8330B. In hindsight, extraction of the entire sample might have yielded a more representative result.

Although MIDAS (2008) provides a rough percentage of the mass of NG typically present in the unfired propellant, the mass and relative percent of NG and ND in the fired propellant is unknown. Combustion of the propellant may result in a greater percentage of NC consumed relative to NG. To evaluate this, 250 mg of 5.56- and 9-mm fired propellant was added separately to 50 mL of DI as well as 50 mL of acetonitrile.

Dissolution of the 5.56-mm propellant in acetonitrile yielded a NG concentration of 350 mg/L, which is equivalent to a mass of 17.6 mg. Assuming the acetonitrile extraction removed the entire quantity of NG from the propellant, this indicates the 5.56-mm propellant is 7.0% NG. Of this 7%, less than 1% (2.3 mg/L or 0.11 mg of NG) was recovered in the batch test DI over a 24-hr period. Similarly, the amount of 2,4-DNT measured in the acetonitrile extract was 2.24 mg/L or 0.11 mg, indicating the propellant was 0.04% 2,4-DNT. No 2,4-DNT was measured in the water extraction, suggesting 2,4-DNT did not leach out of the propellant in 24 hr or the amount leached was not quantifiable with our current instrumentation. Even less 2,6-DNT (0.23 mg/L or 0.01 mg) was found

in the acetonitrile extract, indicating the propellant contains considerably less than 1% 2,6-DNT. As was the case for 2,4-DNT, no 2,6-DNT was observed to have dissolved into the DI.

The concentration of NG in the 9-mm propellant obtained with the acetonitrile extraction was 530 mg/L or 26.5 mg. For 9-mm projectiles, the NG from the fired propellant was comprised of 10.6% of the total mass of the fired propellant. The extraction with DI yielded a NG concentration of 37 mg/L or 1.85 mg, indicating DI extracted 10.6% of the total NG mass within 24-hr. The concentration of 2,4-DNT and 2,6-DNT measured in the acetonitrile extract was 0.2 and 0.3 mg/L, respectively, equal to a 2,4-DNT and 2,6-DNT mass of 0.01 mg. In both cases, this represents less than 1% of the total propellant mass. Neither DNT isomer was detected in the DI extraction of the 9-mm propellant.

Partitioning coefficients were calculated for Test 10d to illustrate how the different processes could contribute to an apparent Kd and to assess the relative magnitude of the contributions comprising dissolution, adsorption and biodegradation (Table 21). Fired propellant with biocide has an average apparent NG K_d of 33 L/kg. The only two processes active in this experiment are adsorption and dissolution.

Table 21. Comparison of apparent adsorption and desorption K_d values for soil, using DI and fresh-fired propellant from Camp Edwards.

Test 10- Evaluation of the Apparent Adsorption K_d for Fresh-Fired Propellant without Biocide				Test 10d - Evaluation of the Apparent Adsorption K_d for Fresh-Fired Propellant with Biocide			
	K_d (L/kg)				K_d (L/kg)		
Replicate	2,4-DNT	2,6-DNT	NG [1]	Replicate	2,4-DNT	2,6-DNT	NG [1]
1	ND*	ND	52	1	ND	ND	34
2	ND	ND	59	2	ND	ND	34
3	ND	ND	64	3	ND	ND	33
Avg	**ND**	**ND**	**58**	**Avg**	**ND**	**ND**	**33**
Test consisted of using 14 g K7 soil with 0.14 g of propellant with no biocide. The propellant contains 11.2% NG and no DNT. This yields a soil concentration of 1120 ppm. Then 70 mL of DI is added.				Test consisted of using 14 g K7 soil with 0.14 g of propellant with biocide added. The propellant contains 11.2% NG and no DNT. This yields a soil concentration of 1120 ppm. Then 70 mL of DI is added.			
*ND – Not detected. See MDL listing in Table 3.							
[1] Note these are calculated values for apparent partitioning ratios and are not K_ds . The apparent K_d includes a component of dissolution, adsorption of the aqueous phase to the soil, and possibly biodegradation.							

Since the K_d of NG for adsorption is already known as approximately 1 L/kg from earlier tests (Table 10), a reasonable assumption is that the majority of this apparent K_d value for fired propellant with biocide is the result of dissolution. Similarly, the fired propellant experiment without biocide resulted in an apparent average K_d of 58 L/kg. The processes active in this experiment are adsorption, dissolution, and biodegradation. If we apportion the contribution from dissolution and adsorption as 33 L/kg (as quantified above), the contribution from biodegradation can be calculated as 58-33 = 25 L/kg. In relative terms, the contribution from each mechanism to the overall fate of NG in this soil is as follows: 1) adsorption ~ 2%; 2) dissolution ~ 55%; and 3) biodegradation ~ 43%. A more rigorous analysis of these competing mechanisms is required to provide more than this simple and qualitative estimate of relative magnitude contributions.

4.14 Nitroglycerin daughter product evaluation

Periodic detections of 1,2-GDN and 1,3-GDN were observed in samples from the batch tests and less often in the column test effluent water. A dozen batch samples with some of the highest observed NG daughter-product detections were selected and re-analyzed with the GC-ECD. HPLC detections of 1,2-GDN and 1,3-GDN were confirmed by GC-ECD (Table 22). In addition, some of the samples reported as below detection limit for 1,2-GDN and 1,3-GDN with the HPLC appear to have detectable levels with the GC-ECD. A formal method detecting limit (MDL) study has not been conducted for the NG daughter products, and the reported values for both the HPLC and the GC-ECD should be considered qualitative.

E4D-YB samples listed in Table 22 were prepared with biocide, which presumably should eliminate biological activity. However, results suggest the presence of NG daughter products. Biological activity was perhaps not completely eliminated with biocide. The results may also suggest reported NG daughter product detections are false positives. Some HPLC detections were not confirmed with the GC-ECD. Biocides may have interfered with these analyses on both the HPLC and GC-ECD, and the detections may represent false positives, as suggested by column test data. Columns without biocide generally had zero to few detections of GDN, whereas columns with biocide (glutaraldehyde) had a higher frequency of detections. This result is the opposite of expectations from a fate perspective. Further method development work for the NG daughter products would be neces-

sary to definitively decide whether these detections are present or are false positives.

Table 22. Comparison of column-analyzed HPLC samples with GC samples analysis.

	GC Analysis (mg/L)		HPLC Analysis (mg/L)	
Sample ID	1,2 GDN	1,3 GDN	1,2 GDN	1,3 GDN
E7 K3-2	0.199 J	0.108 J	BDL	BDL
NB RAIN 3	1.99	1.51	0.91	0.76
E11 - K7 - 1UFD	0.251	0.250 J	0.163	0.113 J
E11 - K7 - 2UFD	0.255	0.213 J	0.207 J	0.148 J
E11 - K7 - 3UFD	0.077 J	BDL	0.118 J	0.080 J
E4D - YB024 – 1	0.223 J	0.161 J	0.290	BDL
E4D - YB048 – 1	0.171 J	0.111 J	0.254	BDL
E4D - YB072 – 1	0.150 J	0.080 J	0.223 J	BDL
E5D – 800 – 1	1.02	0.775	0.972	0.261
E5D – 800 – 2	0.392	0.246 J	0.253	0.068 J
E5D – 800 – 3	0.365	0.228 J	0.238	0.068 J
E6D - J2 – 2	0.228 J	0.120 J	0.158 J	BDL
E4-072-1	1.28	1.69	0.380	BDL
E4-216-1	BDL	1.52	0.350	BDL
E5-800-1	BDL	8.99	0.510	0.120 J
E5-400-1	BDL	4.51	0.290	BDL
E5-100-1	0.332	0.338	0.086 J	BDL
E6-J2-1	1.28	1.28	0.118 J	BDL
E6-E1-1	1.18	1.46	0.122 J	BDL
E6-E2-1	BDL	1.42	0.056 J	BDL
E6-J1-1	BDL	1.34	0.102 J	BDL
BDL = below MDL detection limit, see Table 3, J = estimated value.				

4.15 Mass balance assessment

Thirty-six soil samples from batch tests were analyzed for the concentration of 2,4-DNT, 2,6-DNT, and NG to compare with estimated soil values derived from the aqueous solution at the end of adsorption and/or desorption tests. Each of the 11 samples was analyzed in duplicate (Table 23). The relative % differences (RPD) of the measured and estimated soil concentration were generally less than 30%. The mean RPDs for all 2,4-DNT, 2,6,-DNT, and NG measurements were 18.5%, 17.2%, and 41.4%, respectively. The large RPD for NG is driven by four samples with high RPDs. If these values are excluded, the mean RPD for NG is 24%.

In comparison, the mean RPDs for all 2,4-DNT, 2,6,-DNT, and NG measurements for all experiments were 11.6%, 4.7%, and 3.7%, respectively. The good agreement between the measured and estimated values indicates minimal mass balance loss issues for most samples and validates the use of the aqueous phase measurements to estimate the soil concentration and ultimately calculate K_ds.

Table 23. Comparison of measured and estimated soil concentrations of 2,4-DNT, 2,6-DNT, and NG and calculated RPDs.

Test	Soil Source	Duplicate	Measured Soil Concentration (mg/kg) at Equilibrium			Estimated Soil Concentration (mg/kg) at Equilibrium			RPD		
			2,4-DNT	2,6-DNT	NG	2,4-DNT	2,6-DNT	NG	2,4-DNT	2,6-DNT	NG
Test 3 Ad*	K6		17	15	8.8	17	16	6.8	1	4	26
	K6	Dup	17	15	8.7	17	15	6.7	1	0	26
	J2		16	14	7.7	19	17	7.8	20	20	2
	J2	Dup	16	14	7.7	20	16	8.3	22	16	7
	J1		14	12	6.8	19	17	7.8	31	35	14
	J1	Dup	14	12	7.0	15	12	5.1	5	2	31
	E2		12	9.8	4.4	13	11	5.2	10	8	16
	E2	Dup	12	9.7	4.5	13	11	6.1	15	9	31
	E1		12	10	5.2	13	11	5.1	9	9	2
	E1	Dup	12	10	5.1	13	10	6.2	8	1	21
Test 8 Des**	K2		1.0	BDL	39	1.1	BDL	42	14	0	5
	K2	Dup	1.0	BDL	41	1.1	BDL	42	13	0	1
	K2		1.0	BDL	40	1.1	BDL	42	11	0	3
	K2	Dup	1.0	BDL	42	1.1	BDL	42	6	0	1
	K2		0.9	BDL	36	1.1	BDL	42	18	0	15
	K2	Dup	0.9	BDL	38	1.1	BDL	42	16	0	10
	K1		1.5	BDL	67	1.5	BDL	70	2	0	4
	K1	Dup	1.5	BDL	71	1.5	BDL	70	0	0	1
	K1		1.3	BDL	63	1.5	BDL	70	15	0	10
	K1	Dup	1.3	BDL	65	1.5	BDL	70	12	0	7
	K1		1.3	BDL	61	1.5	BDL	70	15	0	14
	K1	Dup	1.3	BDL	63	1.5	BDL	70	14	0	10
Test 1 Des	K7		12	8	5	21	17	6.1	52	75	24
	K7		12	8	2	13	9.3	3.1	6	11	56
Test 2 Des	K7		40	31	4.2	39	34	7.6	1	7	58
	K7		88	55	19	52	54	33	51	1	200

* Ad – adsorption; ** Des – desorption; BDL – below MDL detection limit., See Table 3.

These are actual measurements prior to the addition of DI. See Table 1.

Table 23 (cont). Comparison of measured and estimated soil concentrations of 2,4-DNT, 2,6-DNT, and NG and calculated RPDs.

Test	Soil Source	Duplicate	Measured Soil Concentration (mg/kg) at Equilibrium			Estimated Soil Concentration (mg/kg) at Equilibrium			RPD		
			2,4-DNT	2,6-DNT	NG	2,4-DNT	2,6-DNT	NG	2,4-DNT	2,6-DNT	NG
Test 3 Des	E1		3.4	4.0	0.4	8.5	7.8	3.5	86	65	162
	J1		6.5	4.1	4.2	8.3	5.9	2.4	24	35	54
	J2		13	8.1	4.7	13	9.7	3.4	2	18	31
Test 4 Des	E3		2.9	1.4	0.1	7.1	8.9	2.6	84	145	184
	E4		1.2	0.6	0.1	4.8	8.4	4.5	120	174	193
Test 10 Des	K7		BDL	BDL	1520	0	0	1120	0	0	32
	K7		BDL	BDL	1760	0	0	1120	0	0	46
	K7		BDL	BDL	2030	0	0	1120	0	0	59
	K7		BDL	BDL	2200	0	0	1120	0	0	67
	K7		BDL	BDL	1970	0	0	1120	0	0	57
	K7		BDL	BDL	1850	0	0	1120	0	0	51
* Ad – adsorption; ** Des – desorption; † BDL – below MDL. See Table 3.											
	Estimated quantity of NG present in the unfired propellant, based on MIDAS.										

5 Column Experiments

Eight column experiments were conducted to assess the adsorption/desorption of NG/DNT under four scenarios: aqueous NG/DNT with biocide (Columns 1A and 1B), aqueous NG/DNT without biocide (Columns 2A and 2B), fresh-fired propellant residue with biocide (Columns 3A and 3B), and fresh-fired propellant residue without biocide (Columns 4A and 4B) (Table 24). Each scenario was evaluated in parallel under aerobic conditions for each of the four set of conditions. Evaluation of this set of column scenarios deviated from the original proposed in the Work Plan (USACE 2007) as outlined in Appendix B.

Prior to conducting NG/DNT loading experiments, a falling-head test was conducted to assess the permeability of the columns. In addition, a tracer test using chloride was conducted to assess the uniformity of the packing of columns and confirm the absence of significant channeling.

Table 24. Column identification and treatment conditions.

Column ID	Treatment Conditions	Cumulative Time (hr)	Cumulative Pore Volumes	Cumulative Volume (L)
1A	Aqueous NG/DNT with Biocide	1,270	111	22.8
1B	Aqueous NG/DNT with Biocide	1,123	100	20.6
2A	Aqueous NG/DNT without Biocide	1,827	157	32.9
2B	Aqueous NG/DNT without Biocide	1,006	88	18.1
3A	Fresh-Fired Propellant Residue with Biocide	1,249	109	22.5
3B	Fresh-Fired Propellant Residue with Biocide	1,081	95	19.5
4A	Fresh-Fired Propellant Residue without Biocide	1,008	88	18.0
4B*	Fresh-Fired Propellant Residue without Biocide	625	109	22.6
* Column 4B was operated at 2x the flow rate of the other columns tested.				

5.1 Column experimental details

5.1.1 Column design

Each of the eight columns was set up in identical fashion (Figure 13). Each column consisted of a glass 5.0-cm inner diameter by 61-cm long chromatography column made by Ace Glass Inc, Catalog #5889-40. The column had a 1-cm permeable glass frit at its base. Two cm of glass wool and 3 cm of 5-mm (70-g) glass beads, which were pre-washed with DI, were placed

on top. The glass wool and beads prevented sediment from clogging the glass frit. Each column was rinsed several times with DI

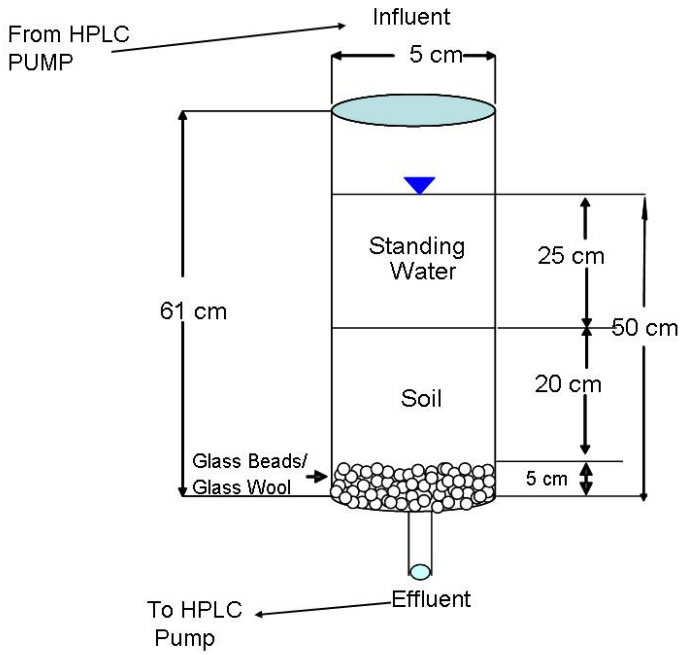

Figure 13. Column design for the study.

prior to placement of the soil within the column. The columns were dry packed, DI was added, and soil was slightly tamped to compress and eliminate voids. A 20-cm (504-g) layer of soil from K6/K7 was placed on top of the glass beads. A mixture of K6/K7 soil was used as there was insufficient volume of K7 remaining for all column tests. A 25-cm column head of water was maintained on top of the soil surface. A stopcock, which is part of the column, was used to control flow. However, the flow rate was widely variable with use of the stopcock. Consequently, columns were operated with the stopcock in the wide-open mode. Effluent flow was controlled by using an 8-multi-head Cole–Palmer Masterflex L/S HPLC peristaltic pump, Model 7519-06. The flow rate for all columns was 0.3 mL/min, except for Column 4B, which was operated with a second Cole–Palmer Masterflex L/S HPLC peristaltic pump, Model 7519-06 at 0.6 mL/min.

The column solution was transferred from the influent reservoir to the column via the HPLC peristaltic pump (Figure 14). Influent solution was added at 0.3 mL/min (0.6 mL/min for Column 4B), maintaining a water column head of 25 cm. The solution then flowed through the column. Ef-

fluent flow was regulated with the HPLC pump and transferred to a Fraction Collector. Two Fraction Collectors, either an ISCO Retriever II Model 69-283-047, or a Spectra-Chrom Model CF-1, were used when collecting samples at a high frequency. When high-sample frequency was not needed, samples were collected manually.

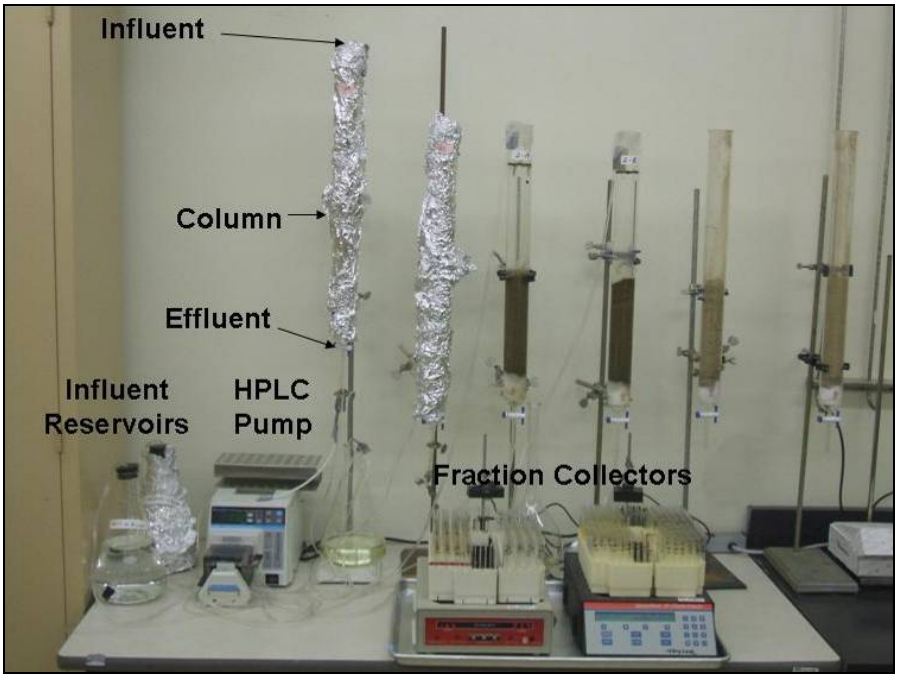

Figure 14. Photograph of experimental set up.

The volume of soil in the column was calculated using the following equation:

2} $$V_{soil} = \pi * r^2 * h$$

where:

V_{soil} = volume of soil in the column (cm^3)
π = pi (3.1416)
r = radius of the column (2.5 cm)
h = thickness of soil in the column (20 cm)

The volume of soil in each column was 392.5 cm^3. Using this information with the porosity of the soil, the residence time of one pore volume can

be calculated for each column. The porosity of the soil can be calculated using:

$$n = 1 - (\rho_b / \rho_s) \quad 3\}$$

where:

n = porosity of the soil (unitless)
ρ_b = bulk density of the soil (1.26 g/cm^3)
ρ_s = particle density (specific gravity) of the soil (2.65 g/cm^3).

The calculated porosity of the soil was 0.52. Because columns typically cannot be compacted as well as field soil, the porosity of a column experiment is usually higher than a field value. Hence, the value of 52% is higher than that associated with the coarse soils at Camp Edwards. The pore volume (P_v) of the soil is the volume of soil (V_{soil}) × porosity (n), which yields a value of 205.9 cm^3. The residence time of one pore volume is then calculated using:

$$R_T = (P_v / Q) \quad 4\}$$

where:

R_T = residence time of one pore volume (s)
P_v = pore volume (205.9 cm^3)
Q = flow rate through the column (0.3 mL/min or 0.005 mL/s)

Solving for the R_T in equation 4 yields a residence time of one pore volume in the column of 4.12E+4 s or 11.4 hr. Because column 4B was operated at a flow rate twice that of others, the residence time of 5.7 hr within the column was half that of the other columns.

5.1.2 Column Conditions

At column start up, a solution of sodium chloride was added with chloride acting as tracer for assessment of flow conditions. The salt solution had a chloride concentration of 100 mg/L for Column 1A and 50 mg/L for the remaining columns. Biocide was added to the solution for Columns 1A, 1B, 3A, and 3B to limit biological activity. The Work Plan (USACE, 2007) originally called for the use of bromide as a tracer (Appendix B). However, a real-time method for monitoring the breakthrough of chloride was de-

sired to allow an appropriate end to the chloride tracer tests, a switch to the NG/DNT mixture, and adjustment of sampling frequency. Use of chloride allowed employment of a field parameter instrument to measure the specific conductance of the soil. However, the background-specific conductance level was elevated in the effluent to such a degree that it largely masked specific conductance because of the addition of chloride to the soil. As a consequence, Columns 1A and 1B were operated without knowing when to expect breakthrough and when to adjust sampling frequency accordingly for the tracer test. Previous research in our laboratory using the same columns, a chloride tracer, and different soil suggested rapid breakthrough and achievement of peak concentration in less than 48 hr. This information was used to guide sampling frequency in the absence of real time data.

The concentration of the reagent-grade, aqueous-spiked solution for the column tests was the same for all experiments (1 mg/L of 2,4-DNT, 2,6-DNT, and NG). The NG and DNT (obtained from Restek, Inc.) were prepared by mixing with DI. Use of an aqueous spiked solution eliminates the possible effects of dissolution. A biocide consisting of glutaraldehyde (1%) was mixed with the influent solution for Columns 1A, 1B, 3A, and 3B. The biocide allowed for potential elimination of all biodegradation phenomena. In the Work Plan (USACE 2007) the use of a mercuric chloride and glutaraldehyde mixture was planned. However, due to waste management issues related to the volume of water containing mercuric chloride, use of this mixture was eliminated from the column experiments (Appendix B). Batch test results, discussed in Section 5, showed that a 1% solution of glutaraldehyde was an effective biocide, and the inclusion of mercuric chloride was not actually needed for the column tests. Combined with use of the aqueous-spiked solution, biocide allows for a strict focus on adsorption. All columns were wrapped in aluminum foil to eliminate the possibility of photo degradation, and the laboratory contained no exterior windows and was generally dark except when samples were collected. Additionally, glass columns were selected because NG can stick to plastic.

If analysis within 24 hr was not possible, the samples were chilled to 4^{o}C until analyzed, in accordance with CRREL procedures. All samples were analyzed with an HPLC, with select samples analyzed on the GC-ECD for the detection of NG daughter products or lower DNT or NG detection limits.

5.1.3 Falling Head Test

A falling-head permeameter test was conducted on each column to assess the hydraulic conductivity of the soil, as suggested by Kulbersh (CENAE). The test consists of allowing unimpeded flow from the column and measuring the change in head over a specified period of time. The equation to perform the test is:

5} $$K = L / t * \ln [h_0/(h_0 - \blacktriangle h)]$$

where:

K = hydraulic conductivity (cm/s)
L = length of the soil sample (20 cm)
t = time (s)
h_0 = height of water column at t = 0
▲h = change in height of water column (cm) after time t

Calculated hydraulic conductivity (Table 25) has to be corrected for changes in viscosity as a function of temperature (the standard is 20°C). In this test the temperature was approximately 17°C, yielding a correction factor of 1.077. Correction factors were obtained from Kasenow (1997). Results indicate that hydraulic conductivity varied from 1.6×10^{-4} to 7.3×10^{-3} cm/s, which is a reflection of the packing consistency of thoroughly mixed soils from the same location (K6 and K7). Still, some variation in hydraulic conductivity may exist due to differential settling as material was transferred from the sample bags to the columns.

Falling-head tests performed on each column indicate that Columns 4A and 4B were less tightly packed than other columns. Overall, the estimated hydraulic conductivity for the soils within the columns was two orders of magnitude higher than estimates for field soil, largely due to an inability to pack soil in a column as tightly as natural soils. Although travel times through the soil column will be different for Camp Edwards soil, if local equilibrium is achieved between the dissolved and adsorbed fractions, the result should be independent of the actual flow rate through the column.

Using information from the current SESOIL model provided by Kulbersh (CENAE), the calculated vertical hydraulic conductivity of the soil at Camp Edwards is approximately 3.12E-5 cm/s over the entire 120-ft thickness of

the unsaturated zone. Depositional lithologic history at Camp Edwards consists of a coarsening upwards sequence. Therefore, soils at or near the surface are expected to have a higher hydraulic conductivity than the average. Additionally, packing soils in a column as tightly as they occur in nature is nearly impossible, and thus column conductivity is likely to be higher than that measured in the field. The measured hydraulic conductivity values presented in Table 25 for the column experiments seem reasonable.

Table 25. Falling-head permeameter hydraulic conductivity determinations.

Column	Change in Water Column height- ▲h (cm)	Time (s)	Uncorrected Hydraulic Conductivity (cm/s)	Corrected Hydraulic Conductivity (cm/s)
1A	18.1	7,200	3.58× 10-3	3.86× 10-3
1B	13	7,290	2.04× 10-3	2.2× 10-3
2A	5.1	7,200	6.34× 10-3	6.83× 10-3
2B	5.4	7,200	6.76× 10-3	7.28× 10-3
3A	2.6	3,600	3.05× 10-4	3.29× 10-4
3B	1.4	3,600	1.6× 10-4	1.72× 10-4
4A	3.2	900	3.8× 10-4	4.1× 10-4
4B	6.5	1,800	8.36× 10-4	9.0× 10-4

5.1.4 Tracer experiments

A tracer test was conducted at the start of each column experiment. All tracer data can be found in Appendix I. The purpose of the tracer test was to determine if the breakthrough curve was consistent with a conservative tracer, i.e. plug-type flow. The effluent curve for a conservative tracer has a nearly vertical rise in concentration (advective dispersive front), which is a plateau near the influent concentration, and once the tracer is removed from the influent, a rapid near vertical decline. Replication of this type of curve would suggest good packing of the soil column with little possible channeling. Dual columns were employed for all tests for each primary test variable evaluated to provide a minimal basis for assessing repeatability.

In Column 1A, an influent chloride concentration of 100 mg/L was used, and in Column 1B a concentration of 50 mg/L was used. The chloride breakthrough curves shown in Figure 15 are consistent with known chlo-

ride behavior. To obtain the minimum necessary volume of liquid for analysis, a sample interval of 30 min was utilized. However, the initial breakthrough of chloride happened so quickly it was not captured. Chloride reached its input concentration in Column 1B in less than 1 day; in Column 1A, it took approximately 1 day. If the breakthrough is too rapid it might be an indication of potential channeling, which may be considered in further analysis of adsorption/desorption test data. Once chloride input was discontinued [approximately 160 hr for Column 1A (~ 14 pore volumes) and 55 hr (~ 4.8 pore volumes) for Column 1B], the chloride rapidly flushed out of the column, i.e. in less than 50 hr (~ 4.4 pore volumes).

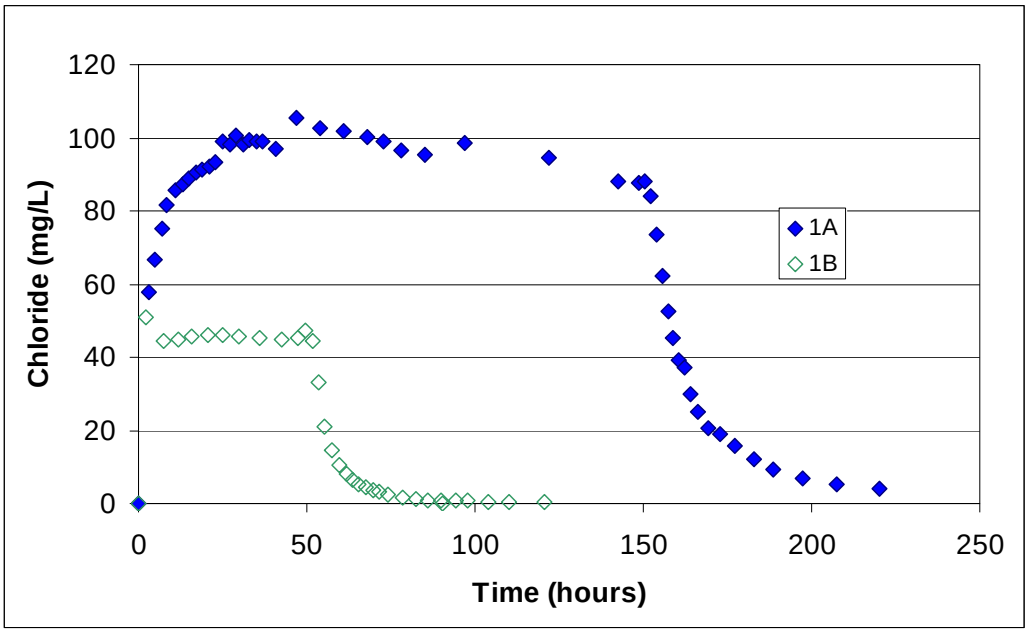

Figure 15. Chloride breakthrough curves for Columns 1A and 1B (aqueous NG/DNT with biocide).

The chloride breakthrough curves for Columns 2A and 2B (subsequently followed by aqueous NG/DNT without biocide) are similar to those observed for Columns 1A and 1B. However, Column 2B results suggested a more gradual rate of chloride breakthrough (Figure 16), suggesting some degree of adsorption of chloride onto the soil. Significant chloride shouldering (Figure 16) may also exist within the first 5 hr for both Columns 2A and 2B. These results are most likely due to non-uniform pore-water velocity, different column packing characteristics, or natural or packing-induced heterogeneity differences. Column 2A chloride levels plateaued near the influent concentration of 50 mg/L in less than 1 day, whereas Column 2B did not plateau until nearly 2 days. At approximately 55 hr the

chloride input was discontinued, and chloride flushed out within 50 hr or at approximately 5 pore volumes for both columns.

The Column 3A and 3B results (subsequently followed by fresh-fired propellant residue with biocide) indicate a classic breakthrough curve for the chloride tracer with influent concentration reached within 48 hr (Figure 17). In both cases, the chloride influent concentration was 50 mg/L. Chloride samples from the flushing phase were not analyzed.

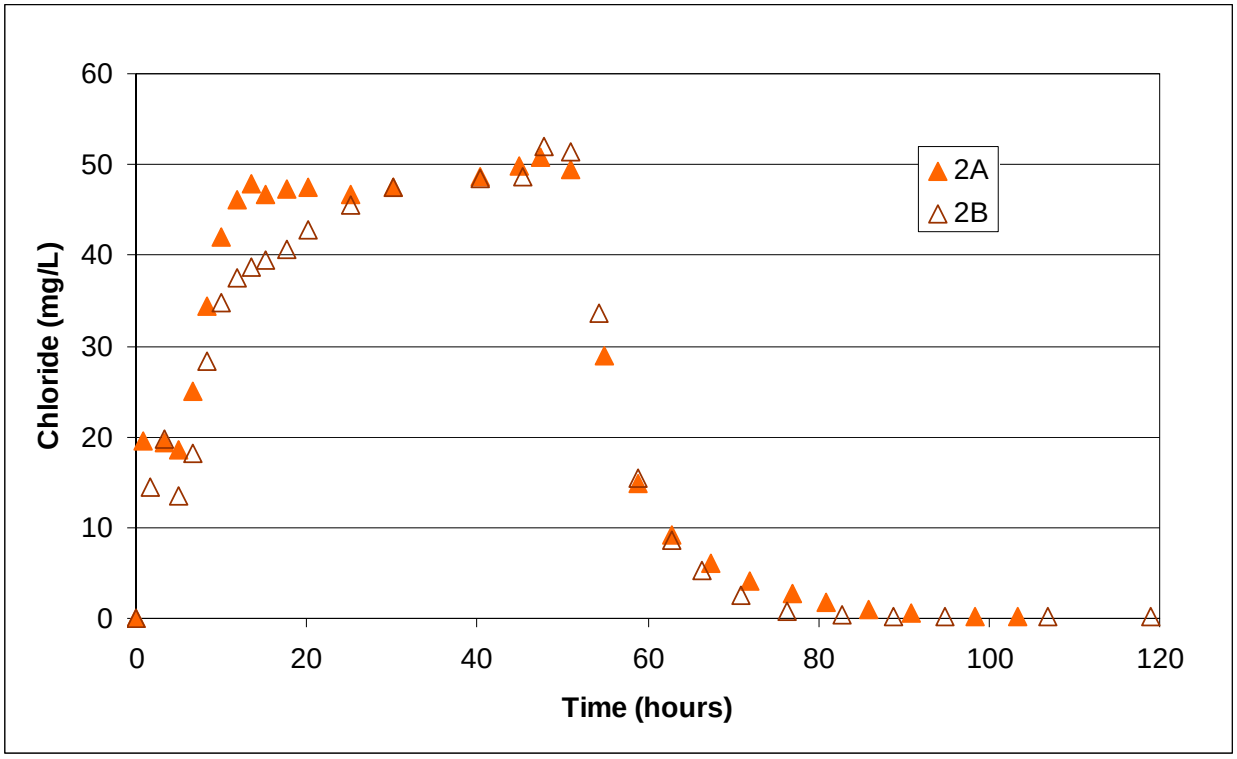

Figure 16. Chloride breakthrough curves for Columns 2A and 2B (aqueous NG/DNT without biocide).

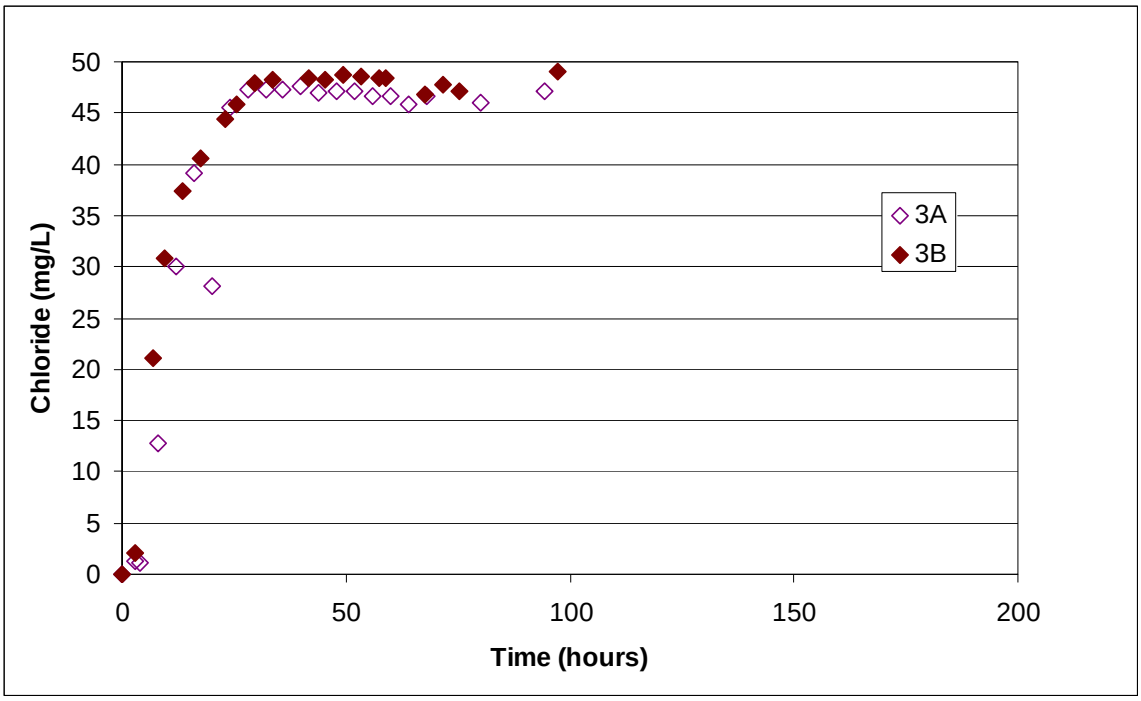

Figure 17. Chloride breakthrough curves for Columns 3A and 3B (fresh-fired propellant residue with biocide).

Similar to the Column 2A and 2B results, initial breakthrough curves of chloride tracer for Columns 4A and 4B (fresh-fired propellant residue without biocide) were,
for the most part, too rapid to measure (Fig. 18). Both Column 4A and 4B chloride breakthrough curves look similar to those of Columns 2A and B. In Column 4B the chloride input was discontinued at approximately 25 hr and at 100 hr for Column 4A. The flow rate for Column 4B was 0.6 mL/min as compared to 0.3 mL/min for all other columns.

During the flushing phase of the tracer test for each of the columns with DI, the slopes of chloride concentration versus time are nearly identical, which suggests the flow conditions in each of the columns was very similar. There does not seem to be any indication of voids resulting in preferential flow within the columns. Interestingly, the columns without biocide (2A, 2B, 4A, and 4B) seem to suggest some possible retardation of the chloride during the adsorption phase of the experiment but not during the flushing phase. The reason for the difference in adsorption chloride-curve

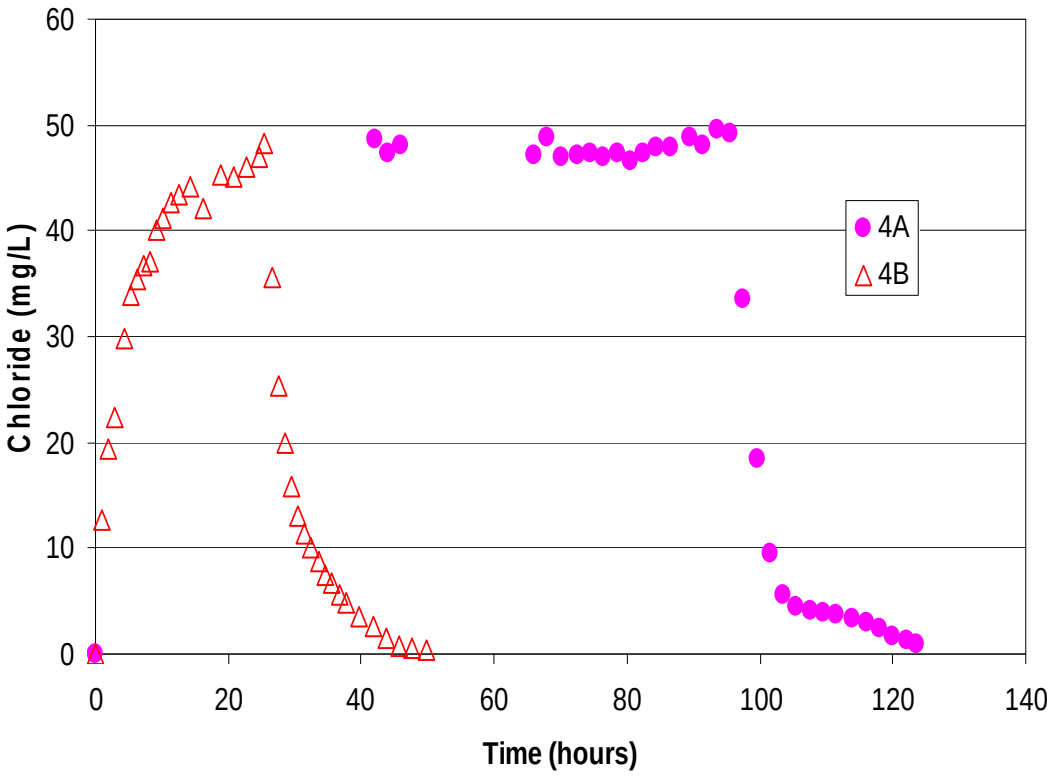

Figure 18. Chloride breakthrough curves for Columns 4A and 4B (fresh-fired propellant residue without biocide).

response for the columns with biocide versus columns without biocide is unknown.

5.2 Aqueous NG/DNT with biocide (Columns 1A and 1B)

Columns 1A and 1B had an aqueous mixture of NG/DNT with biocide (glutaraldehyde) introduced into the soil column in a concentration of 1 mg/L at a flow rate of 0.3 mL/min. Column 1A was operated for a total of 1,270 hr (120 pore volumes) before termination (Table 24). Column 1B was operated for 1,123-hr (100 pore volumes). The Work Plan (USACE 2007)

specified a minimum operation of 960 hr (Appendix B). NG concentrations rapidly increased for the first 24 hr and then increased less rapidly between 24 and 200 hr (Figures 19 and 20; Appendix I). After 200 hr, the concentration of NG appeared to be approaching a maximum concentration, which appeared to be 85 to 90% of the influent concentration. At equilibrium, the effluent concentration should approximately equal the influent, and any difference may be due to excessive time required to attain this level or may be attributed to experimental measurement difficulties. The single biocide application was perhaps not as well mixed or as effective when compared with the dual biocide used in the batch tests. Additionally, some biodegradation may still be occurring. Insufficient $HgCl_2$ biocide was available for the column testing, and thus the single biocide was used. A possible further explanation may be that adsorption/desorption is not linear or perhaps not reversible in the column tests, although this would be quite different from the observed functionality in the batch tests.

Breakthrough of DNTs appears to be slightly slower when compared to NG, with the slope of concentration increase similar to the NG increase between 24 and 200 hr for Column 1A. DNTs appear to have stabilized at 200 hr at a level of 40 to 50% of the influent concentration. This difference is quite significant and it is quite probable that some biodegradation was occurring rather than simply a slow approach to equilibrium. Further evaluation of the data sets requires using software such as BIO1D and/or CXTFIT (See Appendix C, Column Experiments) not currently available for data reduction.

Column 1B results are similar to Column 1A with some slight differences in the trends for NG and DNT. The columns were switched from the adsorption phase (NG/DNT

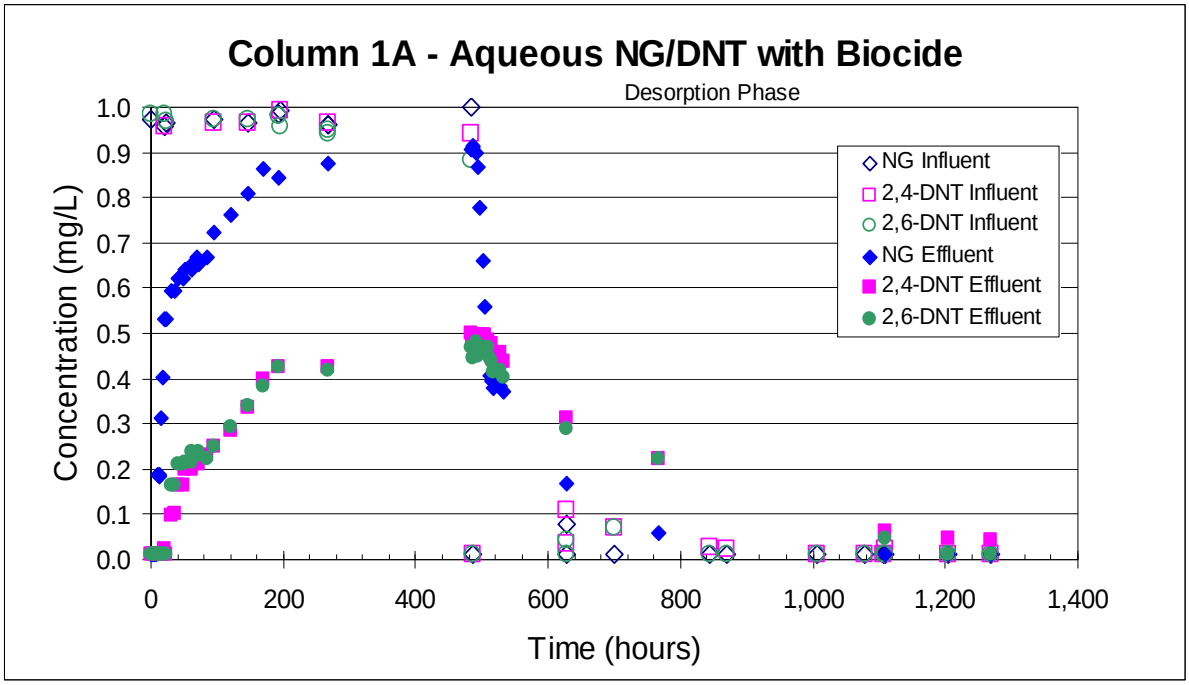

Figure 19. NG and DNT results for column 1A with biocide.

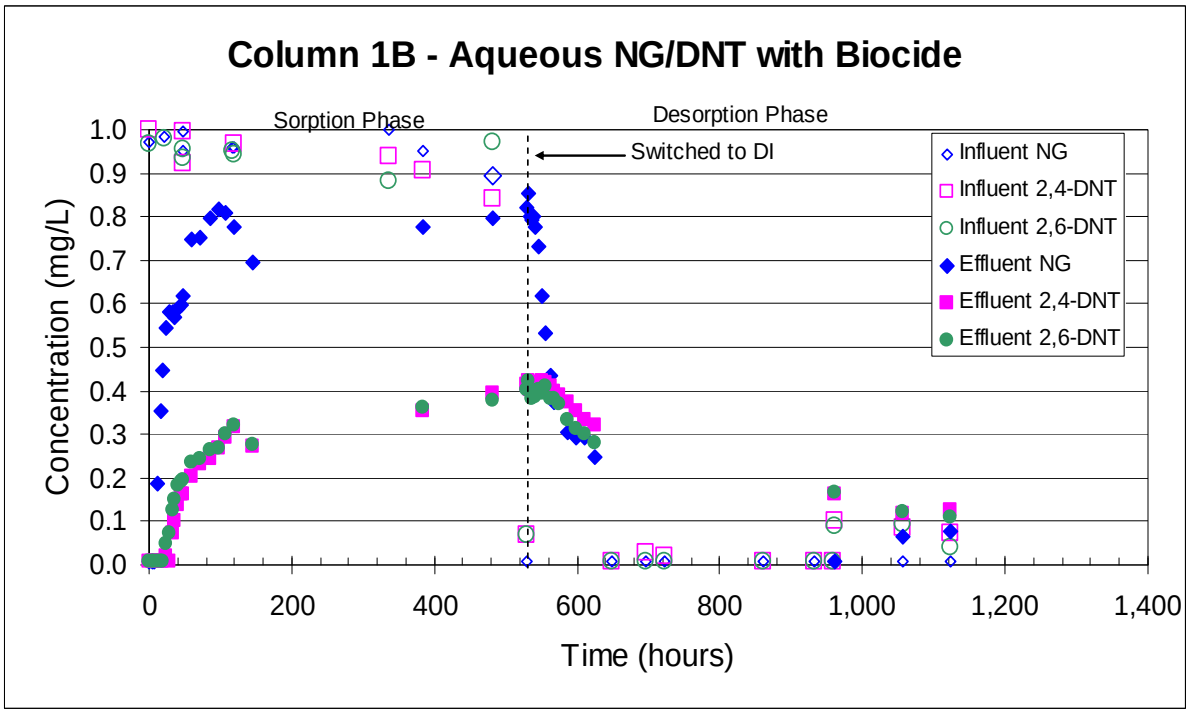

Figure 20. NG and DNT results for column 1B with biocide.

in the influent) to the desorption phase (only DI in the influent) at 485 hr (42.4 pore volumes for Column 1A and 533 hr (46.6 pore volumes) for Column 1B (Appendix I). In both columns, NG levels declined rapidly, whereas DNT decline was less rapid. Column 1A results indicated the sporadic presence of 1,2-GDN at levels less than 0.6 mg/L in the effluent. Column 1B had two detections of 1,3-GDN and several detections of 1,2-GDN in the effluent, at levels less than 0.2 mg/L. Because biocide was used in these columns, biodegradation should not be occurring, and all of the detections are associated with two sample batch runs. A possible explanation is that detections represent false positives. No degradation products were observed in samples analyzed with the HPLC or GC-ECD for Columns 2A or 2B, which contained no biocide.

Adsorption K_d determinations for both columns are provided in Table 26. DNT adsorption K_d values follow the same trend as NG, although they are slightly lower than the NG values. Both NG and DNT values are similar to those estimated by Speitel et al. (2002).

Table 26. Comparison of column 1A and 1B estimated adsorption K_d values with batch experiment values.

	Method for Determining Partitioning Coefficients	Phase	Partitioning Coefficient K_d (L/kg)		
			NG	2,4-DNT	2,6-DNT
Column 1A	VanGenuchten and Wierenga 1986	Adsorption	0.32	13.7	13.7
Column 1B	VanGenuchten and Wierenga 1986	Adsorption	0.43	NA	NA
Batch	Mean of All Data, This Study	Adsorption	0.9	3.2	2.6
Batch	Mean of All Data, This Study	Desorption	1.6	4.9	5.7
Speitel et al. 2002 Batch	Crittenden et al. 2005	Adsorption	1.5	3.3	ND
Speitel et al. 2002 Column	Crittenden et al. 2005	Adsorption	ND	0.5	ND
Speitel et al. 2002 Batch	Crittenden et al. 2005	Desorption	71*	85*	ND
* Value estimated (based on a detection level of 10 ug/L, not a measurement), ND = not determined, NA = not applicable.					

The method used for estimating the adsorption K_d involves an approach developed by van Genuchten and Wierenga (1986), who derived solutions to the advection/dispersion equation. Using the definition of retardation

coefficients (R), the adsorption K_d can be calculated. The value of the retardation coefficient R is estimated using the special solution of the advection/dispersion equation relating the distribution coefficient and fate-and-transport parameters derived by van Genuchten and Wierenga (1986). The value of R is obtained from the graph of concentration ratio versus pore volumes using data from the column studies and is numerically equal to the number of pore volumes at the point at which $C_e/C_o = 0.5$. The equation for determining the retardation coefficient is presented in Appendix C and is discussed in Fetter (1999) and Hounslow (1995).

Effective porosity curves in Chow (1964) show that effective porosity (specific yield) reaches a maximum field value of approximately 35% or 0.35. This is the value for a medium sand. The calculated (effective) value for the porosity of the columns in the column experiment was 52%, which was used in the calculations. Because columns typically cannot be compacted as well as field soil, the porosity of a column experiment is usually higher than a field value.

A calculated correspondence between residence time and pore volumes, based on this effective porosity, is 11.44 hr per volume. The resulting NG K_d value determined with this method for Column 1A is 0.32 L/kg and 0.43 for Column 1B (Table 25). This value is somewhat lower, but generally within a factor of two to three of the batch study values for NG. Column 1A yielded an estimated DNT adsorption K_d of 13.7 L/kg with this approach, assuming the concentration ratio was close enough ($C_e/C_o = 0.5$; Figure 23). Because DNT did not reach a C_e/C_o value of 0.5 for Column 1B, an adsorption K_d value was not estimated by this method.

The column NG data calculated with this method seem to confirm the batch data. Although values were low, both experiments indicated considerable mobility, and the difference in a model-calculated migration, which incorporated the assumption of equilibrium partitioning, would be minimally affected. DNT calculations are less well defined by the simple evaluation because Column 1A values are approximately three times greater than batch values, which were similar to those estimated previously (Spritrl et al. 2002). Additionally, Column 1B data were not available for comparison. Better estimates of retardation coefficients (R) and partitioning coefficients (K_d) from column experiments require software developed for solving the complimentary error function solutions to the advec-

tion/dispersion equation for adsorption/desorption (van Gunuchten and Wierenga 1986).

Several software solutions are available for solving the problem of estimating column parameters and retardation coefficients from laboratory data: 1) BIO1D, available from GeoTrans, Inc. (Srinivasan and Mercer 1988; 2) CXTFIT, available from the U.S. Department of Agriculture (Russell 2008); 3) Visual CXTFIT (Gunnar Nuetzmann 2008); and 4) STAN-MOD, an updated version of the CFITIM code of van Genuchten (Simunek et al. 1999). In the future, some of these methods may be utilized to develop more solutions to the advection/dispersion equation and retardation equation for the experimental data.

Currently, a direct method of calculating a desorption K_d from column experiments without the use of specialized software does not exist (Russel 2008, Nuetzmann 2008). Thus, slopes of the adsorption and desorption curve were compared.

Table 27. Comparison of column adsorption versus desorption slope values.

Test 1A				Test 1B			
	NG	2,4-DNT	2,6-DNT		NG	2,4-DNT	2,6-DNT
Adsorption				Adsorption			
Slope 1	0.0261	0.0065	0.0094	Slope 1	0.0261	0.0065	0.0094
Slope 2	0.0017	0.0017	0.0015	Slope 2	0.0018	0.0015	0.0012
Desorption				Desorption			
Slope 1	-0.0212	NA*	NA	Slope 1	-0.016	NA	NA
Slope 2	-0.0014	-0.001	-0.001	Slope 2	-0.0014	-0.0014	-0.0019
* NA – not applicable.							

Table 27 compares slopes of linear regression equations applied to the aqueous adsorption and desorption data of NG, 2,4-DNT, and 2,6-DNT. Slopes of adsorption and desorption appear to be approximately equal for each phase. Consequently, only retardation should affect the results in the absence of other processes (dissolution, degradation). The slopes are also nearly equivalent across both Column Tests 1A and 1B. From a qualitative perspective (only) the desorption K_d values are approximately equal to the adsorption K_d values and similar to the results obtained from the batch test data. As shown in Appendix C, the slope of the breakthrough curve reflects the dispersivity of the medium (steeper slope, lower dispersivity) and is related to the retarded velocity. This velocity is readily evaluated

when the concentration equals half the influent concentration and the retardation number is related to the number of pore volumes. Both concentration and pore volume are measured values used to determine a 50% breakthrough. The corresponding value of K_d can then be readily estimated, as shown in Appendix C.

The mass-balance determination of NG and DNT for Columns 1A and 1B was based on observed effluent concentrations (Table 28). More than 90% of the NG applied to Columns 1A and 1B was recovered in the effluent. Recoveries of DNT were lower than NG, with values in the 50 to 65% range. The difference may be attributed, in part, to a lack of equilibrium between influent and effluent and also because of incomplete effectiveness of the biocide. To recover a greater amount of DNT, it would have been necessary to operate the columns longer in the desorption mode. Mass-recovery calculations based on the effluent were not possible for other columns because NG and DNT were not detected in the effluent (no breakthrough was observed).

If 10% of missing NG resides in the soil, this would be equivalent to 1 mg of NG and 4 to 5 mg for DNTs. Soil samples were collected from Column 1B in 2-cm lifts, with each lift equivalent to approximately 30 g of soil. Consistent with USEPA Method 8330B, a 10-g subsample was obtained for extraction. Because an aqueous source was used, the soil was not ground up prior to analysis. NG and the DNTs were detected in the soil from Column 1B (Table 29). If the measured concentration is adjusted for the mass of soil in the column, this is equivalent to significantly less than 1 mg of NG and DNT, confirming that very little NG remains on the soil. A higher mass of DNT was also expected to be present on the soil, suggesting the glutaraldehyde biocide was less effective in limiting any microorganism activity capable of degrading the propellants.

Table 28. Mass recovery (%) of NG and DNT from Columns 1A and 1B.

Analyte	Column 1A (%)	Column 1B (%)
NG	92	90
2,4-DNT	63	53
2,6-DNT	59	53

Table 29. Measurement of NG and DNT in soil from Column 1B.

	Concentration (mg/kg)				
Sample ID	2,4-DNT	2,6-DNT	NG	1,2 GDN	1,3 GDN
1B-0-2cm	0.708	0.382	BDL*	BDL	BDL
1B-2-4cm	0.554	0.302	0.062 J**	BDL	BDL
1B-4-6cm	0.64	0.358	0.042 J	BDL	BDL
1B-6-8cm	0.652	0.358	0.06 J	BDL	BDL
1B-8-10cm	0.642	0.364	0.108	0.122	BDL
1B-10-12cm	0.614	0.366	BDL	0.094	BDL
1B-12-14cm	0.69	0.44	0.168	BDL	BDL

* BDL – below MDL, see Table 3.
** J – estimated value below estimated reporting limit.

5.3 Aqueous NG/DNT without biocide (Columns 2A and 2B)

Columns 2A and 2B have the same aqueous mixture of NG/DNT at an influent concentration of 1 mg/L at a flow rate of 0.3 mL/min but with no biocide added. No breakthrough of NG or DNT was observed at the 1 mg/L influent level through 1,827 hr (157 pore volumes) for Column 2A (Table 24; Appendix I).

The Column 2B test was continued for 1,827 hr (157 pore volumes), and no NG or DNT was detected in the effluent up 1,272 hr (111 pore volumes). At 1,273 hr, the influent of Column 2B was increased to a 10-mg/L mixture of NG and DNT, which again resulted in no detections of NG or DNT (Appendix I). At, 1,756 hr (153 pore volumes) the influent concentration was further increased to 100 mg/L. Prior to this time frame, approximately 110 mg of NG and DNT had been applied to the soil column. The last sample collected before stopping the column test yielded detection of NG and NG daughter products. Unfortunately, the column test had to be stopped because the supply of spiking solution was exhausted. The Work Plan (USACE 2007) called for operating the columns for 960 hr or until breakthrough was observed (Appendix B).

In the absence of a biocide, the lack of NG or DNT observed in the effluent at the 1 and 10 mg/L influent concentration for Columns 2A and 2B is likely due to biodegradation. A 100 mg/L solution strength possibly overwhelms or is toxic to soil microorganisms. Further work will be necessary to determine if the degradation capacity of microorganisms has been ex-

ceeded or if high levels are toxic and reduce the microorganisms effectiveness to degrade NG. However, the fact that breakthrough of the DNTs did not occur suggests that microorganisms have a greater capacity for destruction of DNT versus NG. No NG biodegradation products were observed in the effluent at the 1 and 10 mg/L influent level (Appendix I).

One low-level detection of 1,2-GDN, between the MDL and estimated reporting limit, was observed with HPLC analysis at the influent concentration of 1 mg/L. The MDL for the NG daughter products on the HPLC is 0.05 mg/L with the estimated reporting limit at 0.25 mg/L. To evaluate whether one detection and non-detections were valid, a number of column effluent samples were analyzed by GC-ECD. The MDL for the NG daughter products on the GC-ECD was 0.04 mg/L, with the estimated reporting limit at 0.2 mg/L. Because the sample preparation procedures for the HPLC and GC differ, analysis of the same sample was not possible. Therefore, samples closest to the observed HPLC detection were selected and analyzed by GC-ECD. The single HPLC detection was not confirmed by GC-ECD analysis, which has a lower MDL than the HPLC (Table 30). Additionally, the GC-ECD analysis did not reveal any detection of NG daughter products potentially missed by HPLC analysis. Consequentially, this singular HPLC detection was possibly a false positive, and NG daughter products were not generated at quantifiable levels. The NG daughter products are also rapidly degraded.

Table 30. Comparison of column HPLC-analyzed samples with GC sample analysis.

	GC Analysis (mg/L)						HPLC Analysis (mg/L)				
Sample ID	2,4-DNT	2,6-DNT	NG	1,2 GDN	1,2 GDN	Sample ID	2,4-DNT	2,6-DNT	NG	1,2 GDN	1,2 GDN
2A 54/55	BDL*	BDL	BDL	BDL	BDL	2A-59	BDL	BDL	BDL	BDL	BDL
2A 100/101	BDL	BDL	BDL	BDL	BDL	2A-95	BDL	BDL	BDL	BDL	BDL
2A 102/103	BDL	BDL	BDL	BDL	BDL	2A-148	0.022 J	BDL	BDL	BDL	BDL
2A 146/147	BDL	BDL	BDL	BDL	BDL	2A-149	BDL	BDL	0.023 J	BDL	BDL
2A 152/153	BDL	BDL	BDL	BDL	BDL	2A-154	BDL	BDL	BDL	BDL	BDL
2B 52/53	BDL	BDL	BDL	BDL	BDL	2B-48	0.023 J	BDL	BDL	BDL	BDL
2B 138/139	BDL	BDL	BDL	BDL	BDL	2B-120	BDL	BDL	0.028	BDL	BDL
2B 140/141	BDL	BDL	BDL	BDL	BDL	2B-144	BDL	BDL	0.02 J	BDL	BDL
3B 154/155	BDL	BDL	BDL	BDL	BDL	3B-160	BDL	BDL	0.023 J	BDL	BDL
3B 157/158	BDL	BDL	BDL	BDL	BDL	3B-161	BDL	BDL	BDL	0.029	BDL
* BDL – below MDL, see Table 3.											

5.4 Fresh-fired propellant residue with biocide (Columns 3A and 3B)

Columns 3A and 3B tests were constructed and operated in a similar manner to Columns 1A, 1B, 2A, and 2B with the only difference being that 0.25 g of a 50:50 mixture of 5.56 and 9 mm of recently collected (see Section 3.1) solid propellant residue was added to the soil surface. According to MIDAS (2008), the propellant for 5.56 mm and 9 mm consists of 11 and 9.5% NG with no DNT. Therefore, the estimated soil concentration for the top 1 cm, accounting for propellant residue, is approximately 1,000 mg/kg. The Column 3A test was continued for 1,249 hr (109 pore volumes) and Column 3B was operated for 1,081 hr (95 pore volumes). Sporadic low-level detections (< 0.2 mg/L of NG) were observed in the effluent of both columns. One detection of NG occurred at a level of 0.63 mg/L at 24.5 hr in Column 3B (Appendix I). As shown in Table 29, the one low-level detection observed with HPLC was not confirmed with the GC-ECD analysis. Therefore, any detection reported between the MDL and estimated reporting limit for HPLC should not be considered as definitive. These detections are suspected false-positives, which result from interference from the glutaraldehyde. A similar column test conducted without biocide resulted in no detections above the MDL for any compounds.

Soil samples from each 2-cm interval in Column 3B were collected and analyzed. Even after 95 pore volumes, a significant quantity of NG remained in the 0 to 2-cm interval (Table 31). Although it is impossible to determine if NG is sorbed to the soil or in the fired propellant, based on batch test and Column 1A and 1B results, the assumption is that it remains in the propellant residue. Tests with aqueous NG indicate minimal retention of NG onto the soil. The high concentration in the 0 to 2-cm interval is about half the predicted mass, based on the amount of residue applied to the soil surface. However, the actual mass of fired propellant NG used in these experiments is unknown. Of note, for the 0 to 2-cm sample, the entire 35.6 g recovered sample was extracted. The mass balance is based upon an estimate of the mass of unfired propellant obtained from MIDAS (2008).

Soil results from Column 3B also indicate the presence of NG in the soil below the 2-cm interval, which is suggestive of adsorption (Table 31). Although particle migration cannot be excluded, it seems unlikely due to the size of the particles placed on the soil surface. Column 3B was treated with biocide and biodegradation was expected to be eliminated (some biodeg-

radation may continue despite best efforts to sterilize soil with steam and autoclaving).

Table 31. Measurement of NG and DNT in soil from Column 3B.

Sample ID	Concentration (mg/kg)				
	2,4-DNT	2,6-DNT	NG	1,2 GDN	1,3 GDN
3B-0-2cm	BDL*	BDL	518	BDL	BDL
3B-2-4cm	BDL	BDL	100	BDL	BDL
3B-4-6cm	BDL	BDL	1.760	0.066	BDL
3B-6-8cm	BDL	BDL	0.640	0.086	BDL
3B-8-10cm	BDL	BDL	0.472	0.070	BDL
3B-10-12cm	BDL	BDL	0.500	0.080	BDL
3B-12-14cm	BDL	BDL	0.256	0.044 J	BDL
3B-14-16cm	BDL	BDL	0.224	0.062 J	BDL
3B-16-18cm	0.286	0.272	0.420	0.100	BDL
3B-18-20cm	BDL	BDL	0.510	0.078	BDL

* BDL – below MDL, see Table 3.
** J – estimated value below estimated reporting limit.

Table 31 results suggest that some of the unaccounted NG mass may have eluted from the column, even though NG was detected infrequently and at low concentrations. The presence of 1,2-GDN in the effluent suggests some biodegradation of the NG. However, as discussed earlier, the 1,2-GDN detections may be false positives due to glutaraldehyde interference.

5.5 Fresh-fired propellant residue without biocide (Columns 4A and 4B)

Columns 4A and 4B were prepared identically to Columns 3A and 3B except that this test was conducted without biocide added to the influent. Due to pump limitations, Column 4B was operated at twice the flow rate (0.6 mL/min versus 0.3 mL/min) as the other columns. The Column 4A test was ended after 1,008 hr of operation (88 pore volumes) and 4B at 625 hr (109 pore volumes) (Table 24). Although the Work Plan (USACE 2007) called for column operation of 960 hr, because the 4B test was operating at twice the flow rate it was terminated when the test reached a Column 4A near-equivalent pore volume (Appendix B). No NG, DNT or NG daughter products were observed in the effluent samples from either column. The propellant residues utilized presumably only contain NG

(MIDAS 2008). The results from Column 4A and 4B indicate no migration of NG through sorptive and biodegradation processes.

6 Conclusions

NG and DNT undergo adsorption onto Camp Edwards soil with a small portion of both either retarded or irreversibly bound, resulting in desorption K_ds that are two to three times higher than the adsorption K_ds. The K_d derived with aqueous, reagent-grade NG and DNT appear to be reversible and thus are appropriate for modeling purposes. Using all experimental data, the mean adsorption K_d values for all batch tests conducted at room temperature and mixed for 24 hr at a spike concentration of 10 mg/L are 0.9 L/kg for NG, 3.2 L/kg for 2,4-DNT, and 2.6 L/kg for 2,6-DNT, respectively. Similarly, adsorption K_d values were obtained from Columns 1A and 1B for the breakthrough of NG and DNT that were comparable to the batch tests (Table 27). Previous studies with DNT and NG demonstrated adsorption might be a significant mechanism attenuating their movement in the environment (Speitel et al. 2002). Soil adsorption partitioning coefficients (K_d) of approximately 1.5 and 3.3 L/kg were measured for both NG and 2,4-DNT for Camp Edwards soil (Speitel et al. 2002). The results from this study are consistent between the batch and column tests and the earlier work of Speitel et al. (2002).

The DNT values from the present study are similar to reported K_d values for TNT, which is known to sorb to many soil types. Consequently, once dissolved into precipitation, NG has a greater potential to move through soil as compared to DNT. However, the NG K_d is still one to two higher orders of magnitude than that measured for RDX and HMX compounds, which have been documented to move in the environment. Studies (mostly at anti-tank and artillery/mortar firing points) have indicated that NG and DNT migration is limited to several meters (Ogden 2000a, 2000b; Thiboutot et al. 2004; Diaz et al. 2006; Hewitt 2008). This suggests that in addition to adsorption, other mechanisms play a role in limiting NG and DNT movement through the soil column.

Contact time between soil and solution of NG and DNT had no effect on resulting K_d values. Similarly, no apparent difference was found in K_d values for surface soils collected at the three different ranges studied. Results of this study were similar to earlier findings by Speitel et al. (2002) in which soil was acquired in a different area (near the Camp Edwards Central Impact Area). Thus, NG and DNT adsorption K_d values for the other

non-sampled SARs will probably be similar to those obtained at E, J, and K Ranges. No significant differences in K_d value versus soil pH were found. This result is consistent with findings by Haderlein et al. (1996) who determined the DNT K_d to be independent of pH (range 3 to 9 pH). Similar to adsorption results, no desorption K_d value differences were apparent due to variations in soil-to-solution contact time, surface soil sample location, temperature, or pH. As observed in adsorption experiments, slight differences in desorption K_d values were apparent for different soil depths. However, trends are less clear than those for adsorption.

The only variable with a significant effect on adsorption K_d was the depth of the soil sample, which is similar to results of the study by Speitel et al. (2002). Speitel et al. (2002) attributed this difference between surface soil and soil at depth as being related to a decrease in OC. The present study supports this observation. However, CEC may also be a factor. As the depth of the soil sample increased, the desorption K_d values for NG and DNT also decreased. As previously noted in the Adsorption Section, a decrease in OC and/or CEC levels is responsible for a decrease in the desorption K_ds. With lower amounts of OC or CEC, the soil is less capable of retaining NG and DNT. However, as evidenced by the higher desorption K_d numbers when compared to the adsorption K_ds, not all of the NG and DNT is subsequently released. A portion of the NG and DNT may be irreversibly bound to the soil surface.

As expected, the desorption K_d values were somewhat higher than the adsorption K_d values, indicating that once NG and DNT are sorbed to the soil surface, a portion them may be retarded or even irreversibly bound. Because a biocide was used, experimental losses due to biological activity and photo-oxidation processes (columns were wrapped in foil and batch tests were conducted in amber jars) can be ruled out. Therefore, retardation or irreversible binding would explain the higher desorption K_ds as compared to the adsorption K_ds.

As noted in Test 7, some portion of unfired propellant is dissolved and subsequently adsorbed and desorbed from the soil surface. However, in Test 8, which utilized K1 and K2 soils that had been previously contaminated, no NG or DNT was detected in the aqueous phase. Furthermore, NG was detected in the aqueous phase in Test 10, which used fresh-fired propellant. These results suggest that NG and DNT available on the surface of the propellant in the K1 and K2 soils has long since dissolved and

migrated away or biodegraded. The remaining NG and DNT within the propellant are not available for dissolution. These findings demonstrate that aged and weathered propellant in surface soil does not readily dissolve and leach into the subsurface soil. DNT was not present in the propellant, and thus the lack of DNT in the experimental results was expected. These results suggest that availability of DNT or NG from soil measurements cannot be inferred. Because Test 8 was conducted with and without biocide, the lack of NG and DNT in the aqueous phase is not attributable to biodegradation.

Dissolution of NG and DNT is a probable function of how all constituents are incorporated into propellant NC, weathering of the fired propellant, or the effect of firing itself on the propellant prior to subsequent adsorption/desorption processes. The difference in the K_d values from Test 7 and Test 8 results suggests a difference between fresh unfired propellant and weathered fired propellant. Firing at K Range has not occurred within the past year, and any propellant detected in this soil sample has undergone significant weathering. Further considerations related to dissolution are presented in Appendix A.

Clearly, dissolution and degradation are more important processes than adsorption/desorption. To create an effective model, the migration processes of dissolved DNT and NG in the environment during precipitation events must be evaluated.

As expected, breakthrough of the aqueous NG and DNT was observed for the columns with biocide added (1A and 1B). The slope of the concentration increase and maximum concentration appears to be consistent with adsorption values derived during the batch tests. As was observed in batch tests, the soil has a greater affinity for adsorption of the DNTs relative to NG. Approximately 10% of the NG and 50% of the DNTs did not appear in the Camp Edward soil effluent soil, despite application of a glutaraldehyde biocide. No NG and DNT were observed in the effluent from the other columns (no breakthrough). These results suggest that microorganisms present in the soil can readily degrade the available NG/DNT at the aqueous loading rate up to 10 nm/L and/or a soil residue concentration of > 1,000 mg/kg NG at flow rates up to 0.6 mL/min.

The dissolution testing as a separate investigation by Taylor (CRREL) is still in progress and results in Appendix A demonstrate that only about 5%

of the total NG in fired propellant is released during drip tests (dissolution). Almost all of this "available" NG is released within 100 days, and results suggest this material is dissolved from the surface of the propellant grain. Conservative drip rates used to mimic dissolution by precipitation were considerably higher than natural occurrence. Any residual NG remaining in the NC must diffuse very slowly from the bulk interior to the exposed surfaces to allow additional mass transfer to occur during a precipitation event. The current batch and column test results indicate that microbial communities are more than capable of consuming even this greatly accelerated experimental rate of release, and thus the effects of NG/DNT on SARs environments may not be a subject of concern. Although the concentration, which may be toxic to the microbial community, is not known, it is likely to be significantly greater than any quantity released by dissolution on a SAR.

Overall, the results from this study indicate that residual NG and DNT in weathered, fired propellants are not likely to be mobile below the near-surface layers in the environment at Camp Edwards. This is consistent with field observations that show migration of NG to the subsurface aquifers has not occurred or is at de minimis concentrations (DNT) over approximately 12 years of groundwater monitoring. Percent levels of NG were present in the surface at the KD Rocket Range but did not migrate beyond several feet from the surface. Similarly, DNT has been observed in surface soil at the gun and mortar firing points at levels in the low 1,000's of mg/L, but evidence is lacking that demonstrates migration beyond a few feet from the surface.

The findings for Camp Edwards/MMR SARs have application beyond this military installation. The environmental conditions show that contaminant migration appears to be favored for RDX, HMX, perchlorate, and trichloroethylene at MMR. But the lack of migration of NG/DNT in the column experiments, coupled with similar field observations, suggests that NG and DNT are not likely to be mobile at this or other military installations. These results are probably a consequence of the low-discharge rate under dissolution, the low availability of the total for dissolution, reversible adsorption/desorption, and the high rates of degradation processes (biodegradation, photo degradation, phytoremediation) leading to very short half - lives ranging from hours to days.

7 Recommendations

7.1 Use of results

The average adsorption/desorption results obtained in this study can be applied as input terms for the equilibrium (reversible) partition coefficients for NG and DNT in future equilibrium-partitioning modeling activities. If the low partitioning coefficient numbers derived in this laboratory test program were applied directly in unsaturated zone models (for example, SESOIL), although representing retardation through adsorption/desorption during aqueous transport through the soil column, the results would likely indicate a premature impact to groundwater. However, this study suggests that dissolution and biodegradation are two additional fate-and-transport variables that must be considered in any modeling activities. The dissolution studies reported in Appendix A confirm prior expectations that dissolution is slow, and the quantity of NG available for dissolution is only about 5% of the typical 11% of the total fired propellant. In addition, only about 40 to 50% of the total unfired propellant mass in one round is released as fired propellant grains (Appendix A). The balance remaining in the fired propellant after weathering is encapsulated in the hydrophobic nitrocellulose and essentially insoluble, or at least a formidable barrier to dissolution by precipitation.

7.2 Future studies

Dissolution testing continues to better define the limiting diffusion rate from the interior of the grain particles to the exposed surface. There appear to be two dissolution rates indicated by the results of the current study: a comparatively rapid process occurring over 100 days in which about 5% of the typical 5 to 6% NG total as fired (approximately 50% of the original unfired concentration, typically 11% NG (MIDAS 2008); Appendix A, Table A-1), followed by a much slower rate governed by internal diffusion to the exposed fiber/grain surface. This is expected to be a very slow process, which does not impact the microbial capacity to biodegrade available propellant. Additional drip tests are being evaluated by Taylor (CRREL) in a 2-year SERDP project to provide a broader spectrum of results for both propellants and explosives. However, if microbial consumption is capable of eliminating greater rates than is feasible via precipitation

dissolution, the value of such additional testing for simple partitioning investigations for propellant NG/DNT may be moot.

An important question to ask is how much freshly-fired propellant residue loading may be accumulated on the soil surface without resulting in a groundwater impact. In this study, the columns were operated with an equivalent (total) soil concentration of 1,120 mg/kg NG, based on information on unfired propellant in MIDAS (2008). The amount of residue applied to the soil surface could be systematically increased until breakthrough occurs by overwhelming the adsorption sites and the biodegrading capacity of microbial agents. A more practical limit is possibly one of maximum loading, which might result in a toxic concentration to the microbial population, perhaps better defined in biodegradation (microcosm) tests.

Because it appears that biodegradation plays a very important role and additional work is needed to develop a more complete understanding of this process, further consideration may be given to conducting microcosm, batch and column experiments to assess the degree and rate of biodegradation of NG and DNT in Camp Edwards soils.

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Appendix A

Release Rate of NG from Fired Small-Arms Propellants

Susan Taylor, Jen Fadden, and Nancy Perron

INTRODUCTION

Propellant fibers or grains are NC impregnated with either 2,4-DNT (single-base), NG (double-base) or, NG and nitroguanadine [NQ] (triple-base). NG, NQ, and 2,4-DNT are the energetic compounds that are released, and their fate-and-transport are of interest. The amount of energetics available for aqueous transport depends on three processes: 1) how much NG, NQ, or 2,4-DNT deposits on the ground surface after firing different types of guns; 2) the number of rounds expended for each weapon during a training session and the number of training sessions per year; and 3) the rate at which NG, NQ, and 2,4 DNT dissolve from the residues deposited on the ground surface. The form in which the energetic constituents are deposited onto the soil surface has a bearing on how quickly they leach from their NC matrix (Hewitt and Bigl 2005). Since dissolution is thought to be the rate-limiting step preceding biodegradation or aqueous transport, data regarding how quickly NG, NQ, and 2,4-DNT dissolve from NC are needed to determine the flux of these components into the soil. Little work has been done to measure how quickly NG, NQ, or 2,4-DNT leach from propellant NC matrices.

This work was a systematic study of NG release from double-based propellants used to fire small arms. We collected fired and unfired propellants from the W-series propellants used to fire a 9-mm pistol, a 5.56-mm rifle, and a 7.62-mm and 0.50-caliber machine gun. As NG is released when the residue is wet due to rainfall or snow melt, we use laboratory drip tests to mimic field conditions on training ranges where spatially isolated propellant fibers are scattered on top of the soil.

Our data do not predict what occurs to the NG in the soil after the occurrence of dissolution. We purposefully wanted to estimate the amount of energetic that dissolves from the NC matrix. Different models will determine if NG sorbs onto specific soil components or biodegrades. We aimed to provide the rate at which NG dissolves from small arms propellants.

METHODS

Residues were collected on aluminum trays in January 2007 at Camp Ethan Allen, Vermont (Walsh et al. 2007). For the drip tests, we used 39 mg of the fired 9-mm propellant residue, 18 mg of the 7.62-mm residue, 68 mg of the 5.56-mm residue, and 57 mg of the 0.50-caliber residues. Residues were weighed on a Mettler A230 balance and not dried before weighing. The residues were placed in four separate, 1-cm diameter Buchner funnels fitted with a glass frit (Figure A-1a). A syringe pump dripped distilled water (pH 6) at 0.5 mL/hr onto the propellants (Figure A-1b). The water flowed through the frit into a 20-mL scintillation vial. We replaced the vials daily and measured the water volume in the vials. The concentration of NG was measured using a HPLC.

To determine the NG remaining in the fired residue we extracted it in acetonitrile (different volumes were used depending on the residue mass). The acetonitrile and residue were shaken over night and then 1 mL of the acetonitrile was mixed with 3 mL of Milli-Q water and analyzed on the HPLC.

Energetic compounds in the water samples were determined following SW-846 Method 8330B (EPA 2006). Three mL of water was added to the 1-mL acetonitrile extracts and filtered through a 0.45-µm Millipore cartridge. HPLC was used to separate NG and its co-contaminants using a Water NovaPak C8 column eluted at 1.4 mL/min (28°C) with 85:15 water: isopropanol mix and detected by UV at 210 nm. Commercially available standards (Restek), which were developed for energetics, were used for calibration. We prepared 1 and 10 mg/L 8095A standards. Ideally all samples

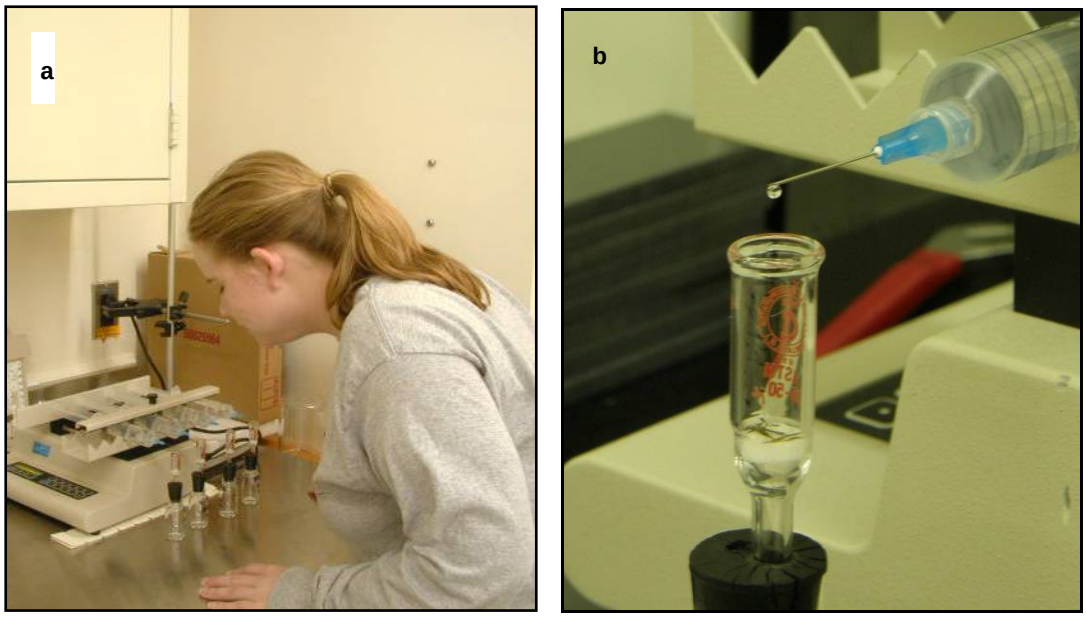

Figure A-1. Laboratory set up for dissolution of propellants.

should have 1 mg/L concentrations, and if these concentrations were > 20 mg/L, they were diluted and reanalyzed. The 1 mg/L standard was run every for 10 samples and was used to recalibrate the instrument. Blanks were tested before each standard run to minimize the possibility of carry over, which would produce a poor calibration. The 10 mg/L standard was interspersed with the samples as an unknown, and a blank was run after each to minimize carry over.

RESULTS

Appearance and composition of propellants

Figure A-2 shows the bullets and casings of the four small arms studied here. Subsequent figures show the unfired and fired propellants. None of the unfired propellant grains have central holes, and their residues appear to be smaller versions of the unfired grains. All of these propellants are coated with graphite to retard the burn rate.

***9-mm pistol**:* The WPR289 propellant used in 9-mm pistols contains ~15% NG (Technical Manual 43-0001-27). The propellant grains vary in size but are typically 0.8 mm in diameter and 0.2-mm thick. Unfired grains are shiny and silvery and often have faceted sides (Figure A-3a). The fired propellants are yellow in color and vary both in size and shape (Figure A-3b). Nine of the unfired grains weighed 2.49 mg or approximately 0.28 mg

each. The fired residues weighed an average of 0.10 mg. When 1 mg of the residue was extracted in 1 mL of acetonitrile and 49 mg of the residue were extracted in 15 mL of acetonitrile, we obtained 0.093 (9% of the residue mass) and 7.8 mg NG (16% of the residue mass) (Table 1).

Figure A-2. Bullets and casings of small arms ammunition.

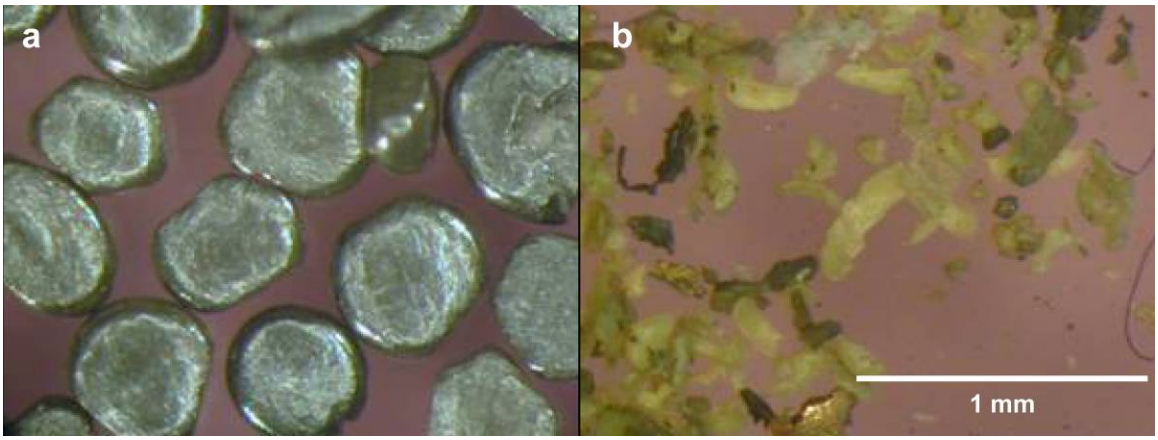

Figure A-3. Unfired grains (a) and fired residues (b), photographed at the same scale, from a 9-mm pistol.

Table A-1. Residues collected and examined to date.

Weapon	Munition	Propellant	Type	% energetics			
				Unfired*	Unfired^	Fired^	
Pistol	9mm	WPR289	NG	12 to 18	12.2±0.6	9	16
Rifle	5.56mm	WC844	NG	9 to 11	9.9±0.2	7	9
MG**	7.62 mm	WC846	NG	8 to 11	10.2±0.3	4	8
MG	0.50 Cal	WC860	NG	8 to 11	9.7±0.1	6	
* From Technical manuals; ^ Analyzed at CRREL, ** MG – machine gun							

5.56-mm rifle: The 5.56-mm rifle propellant examined was the WC 844 formulation that contains ~11% NG (Technical Manual 43-0001-27). The propellant grains showed a range of sizes, typically 1 mm in diameter and 0.3 mm in thickness. The unfired grains were shiny and black (Figure A-4a) whereas the fired propellants were white to yellow in color (Figure A-4b). We weighed four unfired grains individually and obtained an average mass of 0.22 ± 0.09 mg similar to the average mass of 0.20 mg, measured for 23 fired grains. Photographs of the weighed residues show that, in this case, we selected the largest residues to weigh. We estimated the amount of NG still present in the residue by extracting a known mass of the residue in acetonitrile. The NG mass in 2.4 and 53 mg of residue was 0.18 and 4.7 mg of NG, respectively, or about 7 and 9% of the mass of the residues (Table A-1).

***7.62-mm machine gun**:* The WC 846 propellant used to fire the 7.62-mm machine gun contains ~ 9.5% NG (Technical Manual 43-0001-27). These grains are metallic gray in color and are 0.3-mm thick disks that are about 1.0 mm in diameter (Figure A-5a). Four unfired grains weighed 0.91 mg or about 0.23 ± 0.08 mg each.

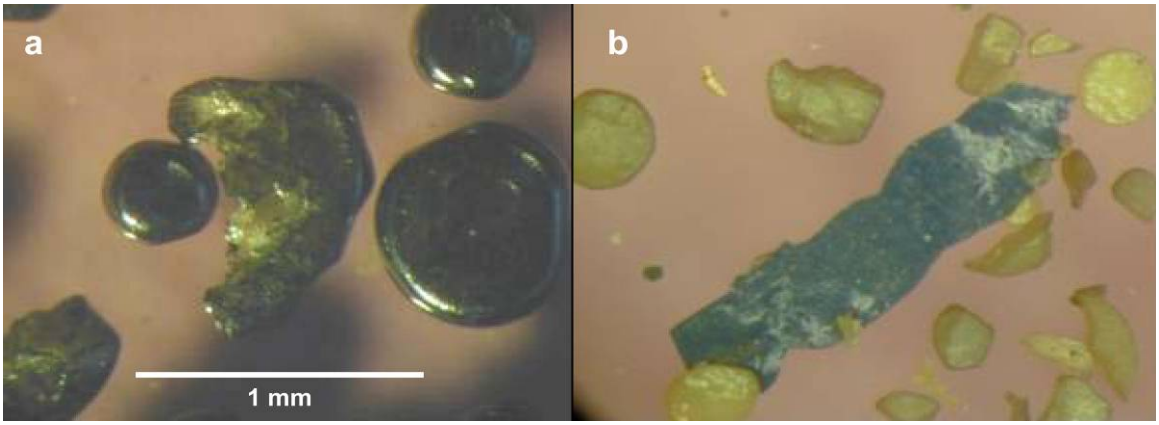

Figure A-4. Unfired grains (a) and fired residues (b) from a 5.56-mm rifle photographed at the same scale.

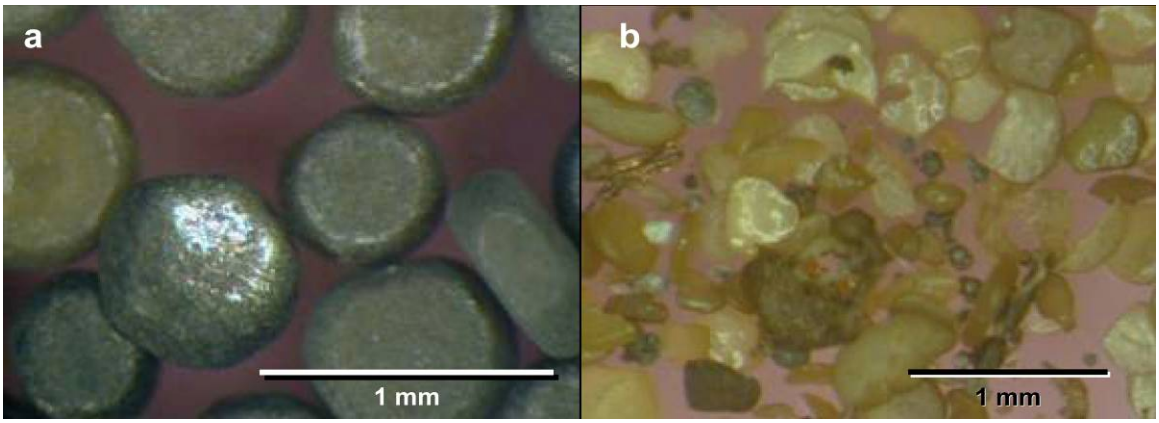

Figure A-5. Unfired grains (a) and fired residues (b) from a 7.62-mm machine gun.

The residues are much smaller than the original grain, generally less than 1 mm in diameter and shiny white to brown in color (Figure A-5b). Twenty-two residue grains weighed 0.09 mg or about 4 µg each. We extracted 0.7 mg of the residue in 1-mL of acetonitrile and 37 mg in 15-mL of acetonitrile. The resulting mass of NG extracted was 0.028 (4% of residue mass) and 3.0 mg (8% of residue mass), respectively (Table A-1).

0.50-caliber machine gun: The WC 860 propellant used to fire the 0.50-caliber machine gun contains ~ 9.5% NG (Technical Manual 43-0001-27). These grains are shiny, black, ~ 0.4-mm thick, and are > 1 mm in diameter (Figure A-6a). We weighed four unfired grains individually on a microbalance and obtained an average mass of 0.57 ± 0.14 mg. Residues are smaller and white-to-brown in color (Figure A-6b). Twenty-eight of these weighed 4.67 mg or approximately 0.17 mg each. We estimated the amount of NG still present in the residue by extracting a known mass of the residue in 1 mL of acetonitrile. For the 0.50-caliber residues, 3 mg of the residue yielded 0.18 mg of NG, or about 6% of the residue mass, which is less than the 8 to 11% NG found in the unfired grains (Table A-1).

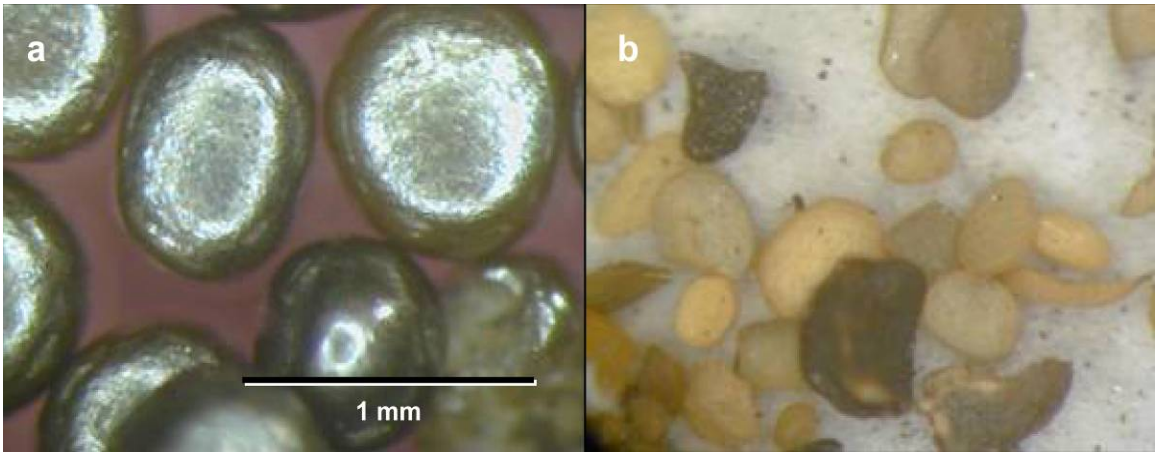

Figure A-6. Unfired grains (a) and fired residues (b), photographed at the same scale, from a .50-caliber machine gun

Dissolution tests

Figures A-7 and A-8 show 100 days of data from the dissolution tests. Figure A-7 plots the cumulative mass of NG recovered in the water samples. We determined that the amount of NG dissolved is correlated with the mass of the residue in the funnel. For example, the amount of fired residue tested (68 mg) was highest for the 5.56-mm and the amount of NG recovered was the largest. Similarly, the mass of the 7.62-mm propellant residue was the lowest, and the amount of NG recovered was the smallest. Because all of these residues are roughly the same size we believed that we would have recovered similar amounts of NG had we used the same mass of propellant in each case. The same data is plotted as the percent of NG recovered, assuming that the residues all contain 10% NG. However, the smaller residue masses have lost a slightly greater fraction of their NG. We think that residues with the least mass will start to 'deplete' their available NG more quickly. Interestingly, all of these residues have lost approximately the same fraction of their NG over 100 days, suggesting similar NG loss rates. Over the course of the 100-day test, the average NG dissolved/day was 0.005% of the NG in the residue.

The results for the drip test conducted on 52 unfired 5.56-mm propellant grains weighing 7.8 mg are shown in Figure A-9. NG was released at a high rate for the first 20 days and then decreased to a much slower rate. The cumulative NG mass loss after 53 days was 0.18 mg. Given that the unfired grains contain about 10% NG (Table A-1) we estimate that collectively the grains contain ~0.8 mg of NG. Because the measured amount of 0.18 mg is

only about 23% of the expected 0.8 mg, the propellant grains must still contain NG. If we assume that the rapid loss of NG is due to contact with water, which given the high solubility of NG in water, (1,250 to 1,950 mg/L), (Rosenblatt et al. 1991, Windholz 1976), seems reasonable, we can estimate the depth to which water has 'extracted' the propellant grain. Assuming the propellant grains possess diameters of ~0.8mm and thicknesses

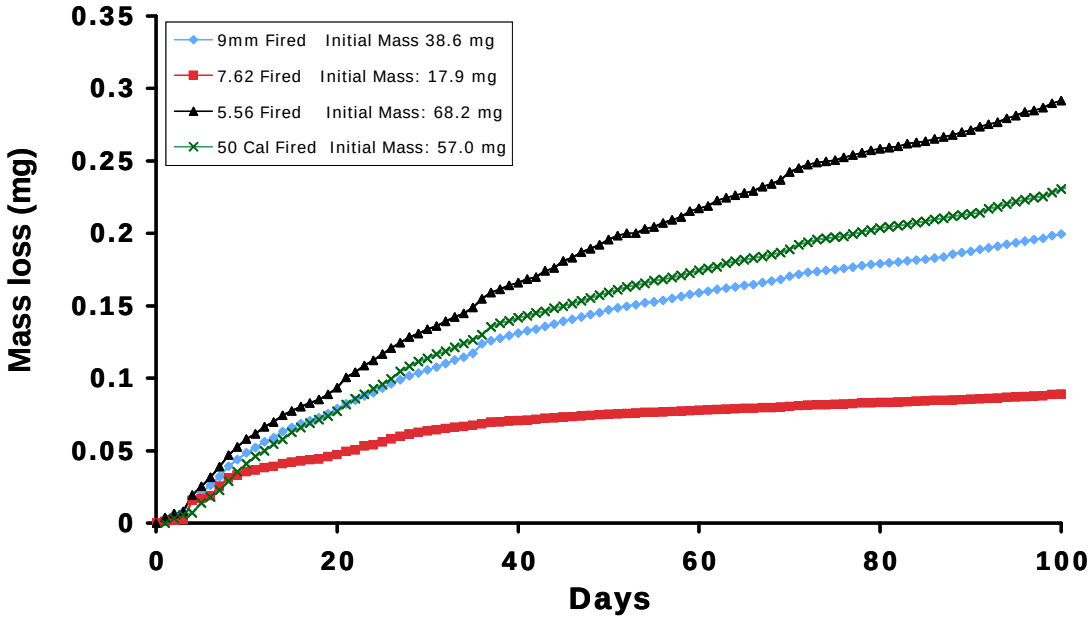

Figure A-7. Cumulative NG recovered from water samples for the four propellant residues.

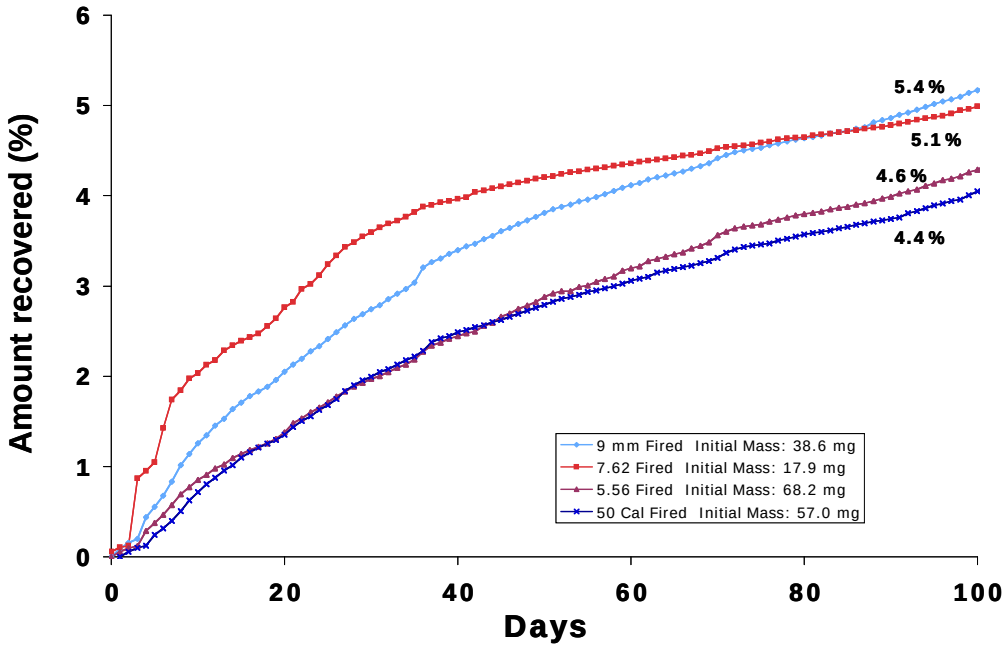

Figure A-8. Percent NG in water samples ratioed to the original amount estimated for the residues (~ 10% of residue mass).

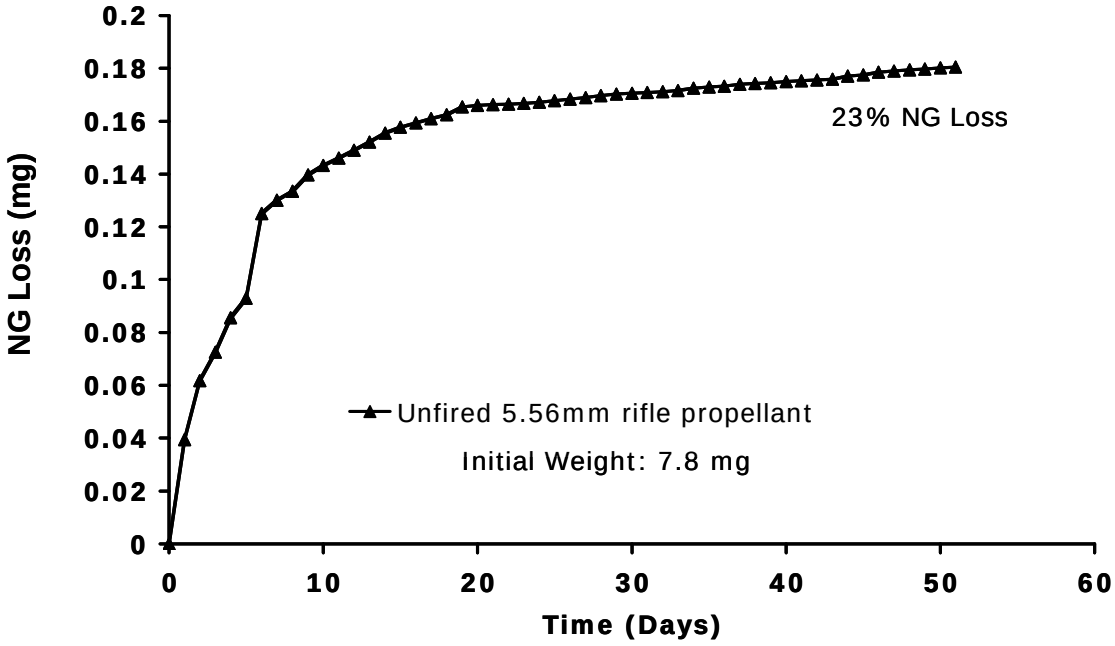

Figure A-9. Cumulative NG recovered from water samples wetting 52 unfired 5.56-mm rifle propellant grains.

of ~ 0.3mm, each will have a volume of ~ 0.15 mm^3. Because collectively, the propellants contain ~ 0.8 mg of NG, individually they have ~ 0.015 mg NG (0.8 mg/52). If each has lost about 23% of its NG (0.004 mg), and the NG is homogeneously distributed in each grain, the loss of NG from the outer 0.05-mm surface of the grain would provide the measured NG.

Discussion

To determine if NG can contaminate groundwater, several pieces of information are required: 1) how much NG is in the soil (NG load); 2) the rate at which NG dissolves from the propellant residue into the water, 3) the rainfall amount, 4) depth to groundwater; and 5) soil properties that affect the adsorption or biodegradation of NG.

NG load

Walsh et al. (2007) measured the amount of NG deposited on clean snow after firing a known number of rounds of small arms (Table A-2). These data, combined with the number of rounds expended for each weapon during the year, provide an estimate of the deposited NG. Alternatively, if range records are not available, one can extract the soil and measure the NG concentration to determine how much NG has accumulated in the soil at the site. Because only a small amount of the chemically-extracted NG is available to be dissolved by rainwater, an NG flux estimation using the chemically extracted value of NG can be calculated in one of the mentioned two ways and constitutes an upper limit.

Table A-2. Summary of NG deposition from Walsh et al (2007).

Weapon	Propellant	Rounds Fired	NG Deposited (mg)	NG/round (mg)	% NG of Round
Pistol- 9mm	WPR289	100	210	2.1	5.4
Rifle- 5.56mm	WC844	100	180	1.8	1.1
MG - 5.56mm	WC844	200	260	1.3	0.8
MG- 7.62 mm	WC846	100	150	1.5	0.6
MG - 0.50 Cal	WC860	200	2200	11	0.7
MG – machine gun.					

Rate of dissolution

After being dripped on for 100 days, the propellant residues still showed fairly constant rates of release, although at a slower rate compared to the first 30 to 40 days (Figure A-8). We estimate that approximately 5% of the

NG in the residue has been lost to date. This rate is slower than that seen in our single experiment on unfired 5.56-mm propellants. Here, the first ~ 20% of NG dissolved within the first few weeks whereas the remaining NG appears to be bound up in the NC matrix and may diffuse out at a slower rate.

Other variables

The rainfall rate, depth to groundwater, and soil type are usually known or can be determined for a site. In this report, Clausen et al. studied how NG in aqueous solution interacts with soil. Holding-time studies using three range soils spiked with NG show rapid disappearance of the compound (Jenkins et al. 2003), suggesting that once in solution and in contact with soil, the half life of NG is short and presumably due to biodegradation.

Given what we know about NG dissolution from propellant residues and its transport parameters, Figure A-10 shows our conceptual model for pathways that could transport NG to groundwater. The most likely pathways are shown with dark arrows.

Because soils differ in their mineralogy, microbiology, and organic content we favor separating the study of dissolution of NG from the propellants from the transport of the dissolved NG. In this approach. the NG leaching from the residues and grains is determined as a function of rainfall rate; column studies using known starting NG aqueous concentrations can then estimate degradation or adsorption of the NG in the soil from the final concentrations. Degradation and adsorption are complex processes that depend on the water path, mineralogy, microbiology, and organic content of the soil, as well as the flow rate through the soil. The question of transport to groundwater becomes tractable only if we can study the dissolution and transport process separately and collectively model them.

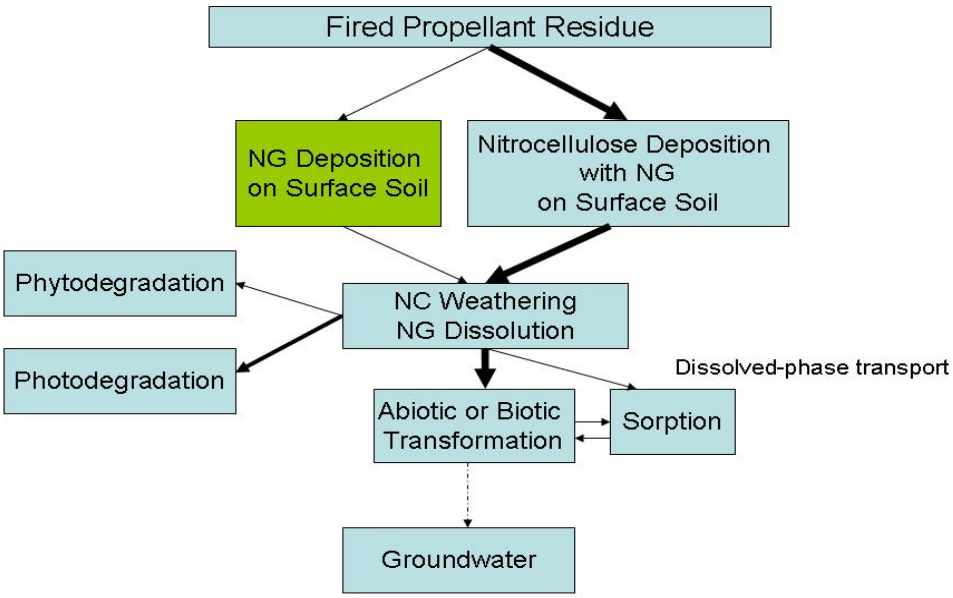

Figure A-10. Conceptual model of how propellant energetics are transported in the environment. Note that thickness of the arrow represents the relative importance of the pathway. Pathways that have not been established or are currently poorly understood are marked with a dashed arrow.

Conclusions

Propellant residues deposited at firing points by small arms studied here are smaller versions of unfired grains and contain NG in a similar percentage as the unfired grains. Our dissolution data suggest loss of the NG from the near surface of the grain is followed by diffusion of the NG to the surface of the grain where it is then dissolved. The interaction of dissolved NG with soil is the subject of the study reported in the main body.

Given that firing deposits about 1% of the NG found present in the round, of this amount only 5% is dissolved after 100 days, and NG appears to biodegrade readily it is not surprising that NG is generally not found in groundwater at SARs.

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Appendix B

Table B-1. Differences between the original Work Plan (USACE 2007) and actual work performed.

Issue	Original Scope as Listed in Work Plan of 11/15/07	Scope of Actual Work Performed	Comment
Number of Samples for Adsorption Experiment	Total samples = 105	Total samples = 175	Pre-test samples included in count as well as additional tests conducted. Previous table counted actual samples and thus numbers were lower because some tests were utilized for multiple experiments.
Chilling versus Freezing of Samples	Freezing of samples prior to analysis	Chilling of samples prior to analysis	A preliminary test was conducted to assess if freezing was necessary or if chilling was adequate to eliminate the need to thaw the samples for several hours after freezing. A concern was that extended thawing might result in degradation of the sample. In addition, chilling greatly increased the efficiency of the sample prep process. As noted, the results indicated no difference between freezing or chilling the samples, and therefore it was not necessary to freeze the samples.
Soil to Solution Ratio	1:20 or 4.5 g of soil	1:5 or 14 g of soil	As noted in the Work Plan the target was for 50% NG/DNT loss. As indicated, preliminary experiments did not result in a 50% loss of NG and DNT. A concentration decrease of 50% was targeted to ensure that the analytical error inherent in the measurement of the initial and final liquid phase concentrations was not the controlling factor in estimating K_ds. Therefore, a soil-to-solution ratio of 1:5 was used, which is consistent with the contingency built into the Work Plan (see page 7).
Soils to be Used in Experiment	Echo Range soil	K7 soil used in experiments	The Work Plan did not identify a specific soil for the tests but used Echo Range as place holder, since the selection of the soil was dependent upon the analytical results. NG and DNT were present in all of the collected surface soil samples (E1, E2, J1, J2, K1, and K2). Therefore, these were deemed unsuitable for the ex-

Issue	Original Scope as Listed in Work Plan of 11/15/07	Scope of Actual Work Performed	Comment
			periments since the presence of NG/DNT in the soil prior to testing could potentially have complicated the determination of the degree of adsorption. Additional surface soil samples were collected and designated (J6, J7, K6, and K7). Since no contaminants (NG or DNT) were detected in the K7 surface soil and its properties are similar to the other soils (Tables 3 and 4) it was selected as the default soil for all tests, except where soil heterogeneity was being evaluated (as in Test 3).
Metals in Soil Samples	Echo Range	K7	As noted above, K7 surface soil was used due to elevated NG/DNT levels in other surface soil samples, which would have made interpretation of the NG/DNT adsorption results difficult. Elevated metal content for copper, lead, zinc, and tungsten in K7 had no bearing on the results (i.e. the K_d values for K7 are the same as those for E1, E2, J1, J2, and K6, which contained less metal).
Test 1. Equilibration Time	Up to 240 hr	Test stopped at 216 hr	As noted, the test was stopped at 216 hr since 240 hr occurred during the weekend.
Test 2. Aqueous Concentrations	0.10, 1, 10, 50, and 100 mg/L	0.10, 1, 10, 40, and 8 mg/L	At the 100 mg/L concentration, a portion of the reagent-grade standard remained as a separate phase and would not completely dissolve in water. Consequently, the maximum concentration level to be evaluated was lowered to 80 mg/L. The 50 mg/L test was reduced to 40 mg/L to be consistent.
Rainwater	No Adsorption Test 7 and Desorption Test 9	Adsorption Test 7 and Desorption Test 9 added	An additional test was conducted with rainwater to see if any effect would occur in the K_d numbers as compared to DI. The purpose was to see how representative the results with DI were when compared to actual field conditions.
Analysis of Aqueous Batch Samples	Single analysis	Duplicate analysis	As a quality check, CRREL researchers decided midway through Test 3 to analyze samples in order on the auto sampler and reanalyze them at the end of the run in reverse order. Each individual sample aliquot was analyzed twice, with each test conducted

Issue	Original Scope as Listed in Work Plan of 11/15/07	Scope of Actual Work Performed	Comment
			in triplicate. There are six results for some individual batch tests.
Analysis of Batch Test Soils	No provision for analysis for NG/DNT in the batch test soils	Post-test analysis of soils for NG and DNT	CENAE directed CRREL to perform this analysis to see if the aqueous phase results agreed with the soil data (i.e. a mass balance check).

Appendix C

Data reduction formulae and methods

Solutes dissolved in groundwater are subject to a number of different processes that can remove them from groundwater. They can be adsorbed onto the surfaces of the mineral grains of the aquifer, sorbed by organic carbon that might be present in the aquifer, undergo chemical transformation (precipitation, abiotic, or biotic degradation), or participate in oxidation-reduction reactions. Because of adsorption processes, some solutes will move much more slowly through the aquifer than the groundwater that is transporting them; this effect is called retardation and will be designated with a retardation coefficient (R). Biodegradation and/or precipitation decrease the concentration of a solute in the plume but may not necessarily slow the rate of plume movement.

The one-dimensional advection–dispersion equation, modified to include adsorption and reaction (degradation), is as follows (Fetter 1993, 1999):

$$1\} \quad \frac{\partial C}{\partial t} = D_L \frac{\partial^2 C}{\partial x^2} - u_x \frac{\partial C}{\partial x} - \frac{\rho_b}{n_e}\frac{\partial C^*}{\partial t} - \left| \frac{\partial C}{\partial t} \right|$$

[dispersion] [advection] [adsorption] [reaction]

where:

C = concentration of solute in the liquid phase (mg/kg)
T = time (s)
D_L = Longitudinal dispersion coefficient (cm^2/s)
x = distance (cm)
υ_x = average linear GW velocity (cm/s)
ρ_b = bulk density of the aquifer (kg/L)
n_e = volumetric moisture content or porosity of the saturated media
C^* = concentration of solute sorbed to the solid (mg/kg)
$|_r$ = chemical reaction-loss terms (biodegradation) (/s).

Sorption processes

Sorption processes include adsorption, chemisorption, absorption, and ion exchange. Adsorption includes the processes by which a solute clings to a solid surface. Ion exchange involves charged particles (for example, oxides and clays). Chemisorption occurs when the solute combines with the surface by a chemical reaction, and absorption occurs when there is diffusion into porous particles.

In this report, adsorption involves a process whereby the solute (NG/DNT) partitions from the aqueous phase onto a particle (mineral and organic matter) and reaches equilibrium between the surface concentration and the solution. The capacity of the soil to remove a solute from the aqueous solution phase is a function of the concentration of the solute. In an experiment where a clean solid is thoroughly mixed with solutions of varying strengths and results are measured and plotted on a graph, the relationship is called an isotherm. At steady state or equilibrium, the shape of the line on a graph (for example, scalar and logarithmic) determines the type of isotherm and has been extensively studied to indicate the properties of the solid and solute.

Equilibrium surface adsorption:

Linear adsorption isotherm. If there is a direct relationship between the amount of solute adsorbed onto the solid {C*} and the concentration of the solute {C} it can be expressed as follows:

$$2\} \qquad C^* = K_d\, C$$

where K_d is an equilibrium partition (adsorption) coefficient (L/kg).

Non-linear adsorption isotherm. There are two common types of non-linear isotherms for situations where equation 2 does not apply:

Freundlich Adsorption Isotherm. This representation is used if the data in equation 2 can be plotted on graph paper and forms a straight line using regression statistics (readily available in Excel). The coefficient K_f is provided by:

$$3\} \qquad C^* = K_f\, C^N$$

$$4\} \qquad \text{Log } [C^*] = \text{Log } [K_f\ ] + N \text{ Log } [C\ ]. \tag{4}$$

Unfortunately, the coefficient is no longer a simple partitioning relationship that is independent of concentration because the relationship can be highly non-linear.

Langmuir non-linear isotherm. The Linear and Freundlich isotherms indicate no limit to the adsorption capacity of the soil, which cannot be true since the number of adsorption sites must be limited in some way whether or not they are charged for ionic inorganic substances or Van de Vaals type for organic substances. Irving Langmuir (1916) introduced another concept to account for this limitation:

$$5\} \qquad C/C^* = 1/[ab] + C/b \qquad \text{(Langmuir isotherm)} \tag{5}$$

where:

a = an adsorption constant related to a binding energy (determined graphically from the slope once b, the intercept, is determined)
b = the constant related to the maximum amount of solute that can be adsorbed by the soil (determined graphically from the intercept).

The value for "1/ab" is the slope, and the value for "1/b" is the y intercept. If the plot is not linear and the line does not go through the origin, the adsorption behavior cannot be explained by simple isotherms (Hounslow 1995).

Note that Test 2 data (Figures 8, 9, and 10) do result in a linear fit with a line intercepting the origin. Because of the uncertainty in the measured concentration with the low-spiked concentration of 0.1 mg/L at the end of the experiment, several of the samples were BDL, and several of the detections were estimated values. These results were ignored in the K_d calculations.

Experimental data reduction

Batch experiments. The ASTM D 4319-83 Standard test Method for Distribution Ratios by the Short Term Batch method provided the basis for the experiments. Measurements of C^* and C are plotted on scalar or logarithmic paper:

Linear isotherm: Plot C^* versus C; Slope = K_d.

Adsorption. As described, an aliquot of soil is thoroughly mixed with an aliquot of solution, which has been previously spiked with a known standard of the investigated solute. In the reported batch tests, 14 g (0.014 kg) of solid were mixed with 70 mL (0.07 L) of various solutions of NG, 2,4 DNT, and 2.6 DNT for sufficient time to reach a steady-state (if not a true equilibrium) final concentration on the solid in intimate contact with the aqueous solution. Pre-testing determined that 24 hr was adequate to reach a steady state (typically this time was < 2 hr, which was the minimum time to prepare for analysis). The final values of C were measured, and C* was either measured or estimated if used in a subsequent desorption experiment. This estimate was readily obtained by calculating the concentration of the solute(s) in the starting and ending solutions, subtracting the corresponding weights of solute, and then dividing that amount by the weight of soil used in the test. Most soils samples were reused in the desorption experiments, but several were used solely for analysis. Partitioning coefficients were calculated using equation 2.

Examples:

To calculate the adsorption K_d the following equation is used

$$6\} \qquad \text{Adsorption } K_d = ((C_o - C_e) * V) / (m * C_e)$$

where:

C_o = the initial spiked solution concentration of NG or DNT (mg/L)
C_{eq} = the measured aqueous concentration at the end of the adsorption test (mg/L)
V = volume of solution used in the experiment (L)
m = mass of soil used in the experiment (Kg).

In our experiments, C_o was usually 8.9 to 9.5 mg/L for each test, V was 70 mL, and m was 14 g.

If we assume that C_o = 10 mg/L, C_e = 7.5 mg/L, V = 0.07 L, and m = 0.014 kg, then the adsorption K_d = ((10 mg/L – 7.5 mg/L) * 0.07 L) / (0.014 Kg * 7.5 mg/L) = 1.7 L/kg.

Desorption. As described, residual solutions resulting from batch adsorption tests were decanted. Soils were air dried prior to placement in sample bottles containing 70 mLs of DI and shaken for 24 hr to reach a steady-state concentration. The final concentration of solutions was analyzed to determine the desorbed quantity of solute, and the final concentration was estimated or measured. Kds were again calculated using equation 2.

To calculate the desorption K_d the following equation is used:

$$6\} \qquad \text{Desorption } K_d = ((m*S_o) - (V*C_{e,d})) / (m*C_{e,d})$$

where:

S_o = estimated soil concentration at the end of the adsorption test (mg/kg)
$C_{e,d}$ = measured aqueous concentration at the end of the desorption test (mg/L)
V = volume of DI used in the experiment (L)
m = mass of soil used in the experiment (kg).

However, before Equation 6 can be used, calculation of the concentration of NG/DNT in the soil at the end of the adsorption test (i.e. the starting soil concentration for the desorption test) is necessary. Because the initial adsorption test spiked-concentration is known, and the measured aqueous phase concentration is known at the end of the adsorption test, the resulting difference is what has been sorbed onto the soil. If this concentration is multiplied by the volume of the water used in the adsorption test and then divided by the mass of the soil, the result is the soil concentration at the end of the adsorption test (S_o).

For example, if it is assumed:

$C_{o,s}$ = the initial spiked solution concentration of NG or DNT =10 mg/L
$C_{e,s}$ = the aqueous concentration at the end of the adsorption test = 7.5 mg/L
V = volume of solution used in the experiment = 0.07 L
m = mass of soil used in the experiment = 0.014 Kg,

an equilibrium NG soil concentration can be determined using the following equation:

7} $S_o = (C_{o,s} - C_{e,s}) * V / m = (10\ mg/L - 7.5 mg/L) * 0.07\ L / 0.014\ kg = 12.5\ mg/kg$.

If it is assumed that $C_{e,d} = 1$ mg/L, which is the aqueous concentration at the end of the desorption test, Equation 8 can be used to calculate the desorption K_d.

8} Desorption $K_d = ((m*S_o) - (V*C_{e,d})) / (m*C_{e,d}) =$

((0.014 Kg * 12.5 mg/kg) - (0.07L * 1 mg/L)) / (0.014 Kg * 1mg/L) =7.5 L/kg.

Residual moisture. Although the soils were air dried after the adsorption test, pore water remained in the soil which contained a residual concentration of NG/DNT, which would affect the accuracy of the calculated K_ds. A decision was made to measure the residual soil moisture and determine if a correction should be applied to the desorption K_ds. This correction was calculated using the final concentration after the adsorption test to estimate the quantity of NG/DNT associated with the pore water, which would directly add to the total initial starting concentration in the soil at the beginning of the desorption test. The final equilibrium concentration in solution would have assimilated this quantity when mixing with the DI occurred.

For example:

9} $W_{diff} = W_{wet} - W_{dry} = 19.5\ g - 14\ g = 5.5\ g \sim 5.5$ mL of solution

where:

W_{wet} = Wet weight of soil = 19.5 g
W_{dry} = Dry weight of soil = 14 g.

In these experiments, the typical difference between wet weight and dry weight was 5.5 g, based on an average of test results of 118 analyzed samples (Appendix D). A calculation is performed to determine how much NG mass is present in the 5.5-mL of residual solution, which is then dried and is present as additional residual NG.

From the above example, it is known that $C_{e,s}$ = 7.5 mg/L at the end of the adsorption test, and thus the NG mass can be calculated using the following equation:

$$10\} \quad NG_{mass} = C_{e,s} * W_{diff} = 7.5 \text{ mg/L} * (5.5 \text{ mL} * 1\text{L} / 1000 \text{ mL}) = 0.04 \text{ mg of NG}$$

To convert to a NG soil concentration, we use the following equation:

$$11\} \quad \delta S_o = NG_{mass} / W_{dry} = (0.04 \text{ mg} / 0.014 \text{ kg}) = 2.9 \text{ mg/kg NG.}$$

The results of Equations 7 and 11 are then added together, which yields a corrected NG concentration of (12.5 + 2.9) = 15.4 mg/kg for the start of the desorption test.

Finally, the corrected desorption K_d = 9.7 L/kg.

As a comparison, the uncorrected desorption K_d = 7.5 L/kg (from equation 8), and the example in Equation 6 yielded an adsorption K_d of 1.7 L/kg.

Column experiments. The adsorption portion of the column experiment is straightforward because the slope of concentration in the effluent, instead of an experimental parameter such as time or pore volume, may be used to assess the K_ds. Software tools are available to assist in evaluation of the data, particularly the retardation coefficient (R), which may be used in conjunction with Equations 1 and/or 2. BIO1D is a simple software tool (Srinivasan 1988), which allows least-squares analysis of data that facilitates a dispersion coefficient D_L to be evaluated (via a Peclet Number P). Another software tool, CXTFIT, is available from the U.S. Department of Agriculture, Agricultural Research Service (Russell 2008), and a spreadsheet version, Visual CXTFIT (Nuetzmann 2008) is also available. These tools utilize a least-squares fit of the data with a solution developed by Van Genuchten and Wierenga (1986):

$$12\} \quad K_d = C^* / C_e$$

where C_e is the final solution concentration in equilibrium with C*,

$$13\} \quad C^* = (C_e - C_o) V / m,$$

where:

C_o = initial concentration of solute
V = volume of liquid
m = weight of soil

$$14\} \qquad R \frac{\partial Ce}{\partial t} = D_L \frac{\partial^2 Ce}{\partial x^2} - \upsilon_x \frac{\partial Ce}{\partial x}$$

This equation was integrated for special conditions {Ce (0,t) =C_o; $\lim_{(x\to\infty)}$ Ce (x,t) = 0}:

$$15\} \qquad C_e\,(x,t)\,/C_o = 0.5\ \mathrm{erfc}\ [\{(Rx) - (ut)\}/\{2(D_L\,Rt)^{1/2}\}]$$

For a finite column length, the value of C_e exiting the column is obtained from:

$$16\} \qquad C_e/\,C_o = 0.5\ \mathrm{erfc}\ [\ \{(P/(4RT)\}^{1/2}\ \{R - T\}\}]$$

where:

T = the number of pore volumes
P = the Peclet Number: P = $\upsilon h/D_L$
h = soil column length
erfc = the complementary error function readily available in standard mathematical texts (Fetter 1999).

Equation (16) has a simple solution at R = T, as erfc [0] = 1 and C_e / Co = 0.5, which states that the retardation coefficient (R) in a column study is approximately equal to the number of pore volumes (T) required to achieve a 50% breakthrough. The distribution coefficient (K_d) for a linear isotherm is then readily calculated from equation (17).

For experimental situations where the ratio of Ce / Co = 0.5 does not co-operate (see experimental data graphs for 2,4 DNT and 2,6 DNT) (Figures 19 and 20), the least-squares analysis afforded by CXTFIT is required to be performed so that experimental data are statistically regressed to match the actual concentration ratio with the number of pore volumes (T) and the corresponding value of R, R_f or R_{fl}, so that equations 17 ,18 0r 19 may be solved for $K_{d,f}$ or $(a, b)_{fl}$.

$$17\} \qquad R = 1 + (\rho_b * K_d\ ) / n_e \qquad \text{(Linear Isotherm)}$$

18} $R_f = 1 + (\rho_b * K_f * NC^{N-1}) / n_e$ (Freundlich Isotherm)

19} $R_{fl} = 1 + \rho_b / n_e [ab/(1 + aC)^2]$ (Langmuir Isotherm)

Note that only K_d is an equilibrium partition coefficient in the sense of definition of the linear isotherm and is measured as L/kg. The Freundlich coefficient has a concentration dependence, and the Langmuir expression does not have an equilibrium partition expression, although the product (ab) does have units of L/kg.

Desorption experiments: No direct method is available to calculate desorption K_d in column experiments. Methods have been developed based on statistical evaluations of the advection/dispersion equation as outlined above. However, as discussed in Section 5.2, the adsorption and desorption slopes for Columns 1A and 1B were compared to obtain a qualitative assessment of whether the desorption K_d was more, less, or equal to the adsorption K_d.

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Appendix D

Evaporated water mass at end of adsorption test.

Sample Number	Wet Weight (g)	Dry Weigh (g)	Difference (g)
E7-K4-1	136.5	135.8	0.7
E7-K3-C	137.2	135.9	1.3
E7-K4-3	140.6	135.9	4.7
E7-K5-2	140	137.2	2.8
E7-K5-C	143.2	137.4	5.8
E7-K3-2	140.1	136	4.1
E7-K5-1	139.3	135.9	3.4
E7-K5-3	139.1	137.3	1.8
E7-K3-1	140.5	135.7	4.8
E7-K4-C	140.8	136.2	4.6
E7-K4-2	140.6	135.6	5
E7-K3-3	142	137.3	4.7
E6-J2-B	140.2	135.8	4.4
E6-K6-B	144.2	136.5	7.7
E6-E1-2	139.6	134.9	4.7
E6-E1-B	139	134.7	4.3
E6-J2-2	144.6	135.7	8.9
E6-K6-2	142.7	136.8	5.9
E6-J1-2	140.8	137.4	3.4
E6-J1-B	140	137.3	2.7
E6-E2-2	138.2	137.3	0.9
E6-E2-B	138	134.9	3.1
E4D-4B120-1	140.9	134.4	6.5
E4D-4B120-2	142.3	135.9	6.4
E4D-4B120-3	141.8	135.7	6.1
E7-E3-1	140.4	136.1	4.3
E7-E3-2	139.5	135.6	3.9
E7-E3-3	139.7	135.5	4.2
E7-E3-B	140.7	136.7	4
E7-E4-1	140.5	137.2	3.3
E7-E4-2	140.6	137.5	3.1
E7-E4-3	138.5	135.7	2.8
E7-E4-B	139.5	136.5	3
E7-E5-1	140.2	136.9	3.3
E7-E5-2	139.1	135.8	3.3

Sample Number	Wet Weight (g)	Dry Weigh (g)	Difference (g)
E7-E5-3	139.7	136	3.7
E7-E5-B	140.2	136.8	3.4
E7-J3-1	150.6	144.9	5.7
E7-J3-2	151.3	146.7	4.6
E7-J3-3	151.5	146.1	5.4
E7-J3-B	150.1	145.3	4.8
E7-J4-1	149.9	144.8	5.1
E7-J4-2	150.8	145.4	5.4
E7-J4-3	151.1	145.9	5.2
E7-J4-B	149.9	144.8	5.1
E7-J5-1	148.4	145.1	3.3
E7-J5-B	147.9	144.4	3.5
E6ACN-E1-024-3	137.9	135.2	2.7
E6ACN-E2-024-3	140.8	137.6	3.2
E6ACN-J1-024-3	138.8	135.6	3.2
E6ACN-J2-024-3	140.6	137.3	3.3
E6ACN-K6-024-3	140	136	4
E10-K1-1d-YB	146.1	137.8	8.3
E10-K1-2d-YB	145.9	138.8	7.1
E10-K1-3d-YB	144	137.6	6.4
E10-K2-1d-YB	146.4	138.7	7.7
E10-K2-2d-YB	144.7	137.6	7.1
E10-K2-3d-YB	145.1	138.3	6.8
E7D-E3-1	139.9	136.1	3.8
E7D-E3-2	139.4	135.5	3.9
E7D-E3-3	139	135.4	3.6
E7D-E3-B	140.4	136.7	3.7
E7D-E4-1	140.2	137	3.2
E7D-E4-2	140.5	137.4	3.1
E7D-E4-3	139.1	135.6	3.5
E7D-E4-B	139.9	136.5	3.4
E7D-E5-1	140.3	136.8	3.5
E7D-E5-2	138.7	135.8	2.9
E7D-E5-3	139.7	136	3.7
E7D-E5-B	140.1	136.8	3.3
E7D-J3-1	148.8	144.8	4
E7D-J3-2	150.8	146.6	4.2
E7D-J3-3	150.8	146	4.8
E7D-J3-B	149.8	145.4	4.4
E7D-J4-1	149.5	144.7	4.8

Sample Number	Wet Weight (g)	Dry Weigh (g)	Difference (g)
E7D-J4-2	150.4	145.4	5
E7D-J4-3	150.7	145.9	4.8
E7D-J4-B	149.8	144.8	5
E7D-J5-1	148.5	145	3.5
E7D-J5-B	147.9	144.3	3.6
E9D-PH9-1	146.1	136.5	9.6
E9D-PH9-2	145.4	136.1	9.3
E9D-PH9-3	144.9	135.9	9
E9D-PH9-B	146.6	137.4	9.2
E7D-J5-2	141.7	137.6	4.1
E7D-J5-3	142.1	138.4	3.7
E12D-K7-NB-1-rain	144.9	136.2	8.7
E12D-K7-NB-2-rain	147.3	138.7	8.6
E12D-K7-NB-3-rain	147.1	137.7	9.4
E12D-K7-NB-B-rain	147.3	138.4	8.9
E12D-K7-YB-1-rain	147.1	139.1	8
E12D-K7-YB-2-rain	148	139.7	8.3
E12D-K7-YB-3-rain	147	138.5	8.5
E12D-K7-YB-B-rain	147	139.3	7.7
YB-rain-B	144.7	136.4	8.3
NB-rain-B	144.9	136.9	8
YB-rain-C	123.1	122.7	0.4
NB-rain-1	144.5	136.1	8.4
NB-rain-2	150.8	136.4	14.4
NB-rain-3	144.2	136.6	7.6
NB-rain-C	123.5	122.7	0.8
YB-rain-1	144.3	136.6	7.7
YB-rain-2	144.8	137	7.8
YB-rain-3	145.4	137.2	8.2
T12A-K6/7-R1	146.7	137.8	8.9
T12A-K6/7-R2	146.3	135.8	10.5
T12A-K6/7-R3	146.4	137.2	9.2
T12B-K6/7-R1	144.9	136.5	8.4
T12B-K6/7-R2	143.9	136	7.9
T12B-K6/7-R3	144.2	136.7	7.5
YBD-rain-B	144.7	136.4	8.3
NBD-rain-B	144.9	136.9	8
NBD-rain-1	144.5	136.1	8.4
NBD-rain-2	150.8	136.4	14.4
NBD-rain-3	144.2	136.6	7.6

Sample Number	Wet Weight (g)	Dry Weigh (g)	Difference (g)
YBD-rain-1	144.3	136.6	7.7
YBD-rain-2	144.8	137	7.8
YBD-rain-3	145.4	137.2	8.2
		Mean	5.5
		Std Dev	2.6

Appendix E

Quality assurance/quality control

A series of quality assurance/quality control checks were performed on the HPLC results to check the accuracy and precision of the data. The first consisted of an analysis of 1 mg/L calibration standard containing 2,4-DNT, 2,6-DNT, NG, 1,2-GDN, and 1,3-GDN, with each batch test. A 1-mg/L calibration standard was run for every 10 samples. The results indicate good accuracy (Table E-1). In addition, a multi-level MDL calibration study was conducted and documented in Hewitt et al. (2008). Because the experiment consisted of a known spike solution and was essentially a matrix spike, matrix spike and matrix spike duplicate samples were not prepared.

Table E-1. Results of 1 mg/L calibration standards containing NG, 2,4-DNT, 2,6-DNT, 1,2-GDN, and 1,3-GDN.

Test Number	Sample #	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	1,2 GDN (mg/L	1,3 GDN
1	NG 2,4-DNT 2,6-DNT DiNGs 1 ppm	1.000	1.105	1.040	1.026	1.031
2	NG 2,4-DNT 2,6-DNT DiNGs 1 ppm	0.997	0.998	0.998	0.997	1.006
3	NG 2,4-DNT 2,6-DNT DiNGs 1 ppm	0.998	1.010	1.006	0.981	1.003
4	NG 2,4-DNT 2,6-DNT DiNGs 1 ppm	0.997	0.998	0.998	0.997	1.006
5	NG 2,4-DNT 2,6-DNT DiNGs 1 ppm	1.0	1.0	1.0	0.99	0.99
6	NG 2,4-DNT 2,6-DNT DiNGs 1 ppm	0.980	0.984	1.008	0.999	0.994
7	NG 2,4 - DNT 2,6 DNT DiNGs 1ppm	1.002	1.001	1.005	1.000	1.006
Average Concentration		0.995	1.012	1.008	0.998	1.005

During each batch test, a blank sample was prepared for each variable evaluated (i.e. for every individual batch test run in triplicate there was an associated blank sample). The blank sample consisted of 70 mL of DI added to the sample jar that remained with the batch samples through the duration of the test, sample preparation, and analysis. The results provided in Table E-2 are data provided by the instrument, which in some cases indicate a detection. However, a review of chromatograms for all potential detections indicated a peak close to the retention time for the target analyte but not close enough to be the target analyte.

Table E-2. Blank results from the batch test in order of analysis run.

Test Number	Vial Number	Forward/ Reverse	Sample Number	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	1,2 GDN (mg/L)	1,3 GDN (mg/L)
8		Forward	E10ACN-YB-Blank	BDL	BDL	BDL	BDL	BDL
4		Forward	E7D-E9D-Blank	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7D-E9D-Blank	BDL	BDL	BDL	BDL	BDL
8		Reverse	E10ACN-YB-Blank	BDL	BDL	0.058 J	BDL	BDL
8		Reverse	E10-Blank-YB	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7D-Blank	BDL	BDL	BDL	BDL	BDL
10		Forward	E12D - NB-Blank	BDL	BDL	BDL	BDL	BDL
10		Reverse	E12D - NB-Blank	BDL	BDL	BDL	BDL	BDL
10		Forward	E12D - YB-Blank	BDL	BDL	BDL	BDL	BDL
10		Reverse	E12D - YB-Blank	BDL	BDL	BDL	BDL	BDL
1	A09	Forward	E4D-YB-B	BDL	BDL	BDL	BDL	BDL
2	A10	Forward	E5D-BLANK	BDL	BDL	BDL	BDL	BDL
7	A27	Forward	E11-K7-Blank	BDL	BDL	BDL	BDL	BDL
7	A27	Reverse	E11-K7-Blank	BDL	BDL	BDL	BDL	BDL
2	A10	Reverse	E5D-BLANK	BDL	BDL	BDL	BDL	BDL
1	A09	Reverse	E4D-YB-B	BDL	BDL	BDL	BDL	BDL
4		Forward	E7-E3-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7-E3-B	BDL	BDL	BDL	BDL	BDL
4		Forward	E7-E4-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7-E4-B	BDL	BDL	BDL	BDL	BDL
4		Forward	E7-E5-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7-E5-B	BDL	BDL	BDL	BDL	BDL
4		Forward	E7-J3-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7-J3-B	BDL	BDL	BDL	BDL	BDL
4		Forward	E7-J4-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7-J4-B	BDL	BDL	0.220 J	BDL	BDL
4		Forward	E7-J5-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7-J5-B	BDL	BDL	BDL	BDL	BDL
5		Forward	E8D-T12-B	BDL	BDL	BDL	BDL	BDL
5		Reverse	E8D-T12-B	BDL	BDL	BDL	BDL	BDL
5		Forward	E8D-T32-B	BDL	BDL	BDL	BDL	BDL
5		Reverse	E8D-T32-B	BDL	BDL	BDL	BDL	BDL
8		Reverse	E10-Blank-YB	BDL	BDL	BDL	BDL	BDL
6		Reverse	E9-PH9-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7D-E3-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7D-E5-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7D-E4-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7D-Blank	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7D-J5-B	BDL	BDL	BDL	BDL	BDL

Test Number	Vial Number	Forward/ Reverse	Sample Number	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	1,2 GDN (mg/L)	1,3 GDN (mg/L)
4		Reverse	E7D-J3-B	BDL	BDL	BDL	BDL	BDL
4		Reverse	E7D-J4-B	BDL	BDL	BDL	BDL	BDL
3		Reverse	E6ACN-Blank	BDL	BDL	BDL	BDL	BDL
1		Reverse	E4D-240-B	BDL	BDL	BDL	BDL	BDL
8		Reverse	E10ACN-Blank	BDL	BDL	BDL	BDL	BDL
8		Forward	E10ACN-Blank	BDL	BDL	BDL	BDL	BDL
8		Forward	E4D-240-B	BDL	BDL	BDL	BDL	BDL
3		Forward	E6ACN-Blank	BDL	BDL	BDL	BDL	BDL
9		Forward	E12 -K7-NB-B-rain	BDL	BDL	BDL	BDL	BDL
9		Reverse	E12 -K7-NB-B-rain	BDL	BDL	BDL	BDL	BDL
9		Reverse	E12 -K7-YB-B-rain	BDL	BDL	BDL	BDL	BDL
3		Forward	E6D-E1-B	BDL	BDL	BDL	BDL	BDL
3		Forward	E6D-E2-B	BDL	BDL	BDL	BDL	BDL
3		Forward	E6D-E7D-B	BDL	BDL	BDL	BDL	BDL
3		Forward	E6D-J1-B	BDL	BDL	BDL	BDL	BDL
3		Forward	E6D-J2-B	BDL	BDL	BDL	BDL	BDL
3		Forward	E6D-K6-B	BDL	BDL	BDL	BDL	BDL
3		Reverse	E6D-E1-B	BDL	BDL	BDL	BDL	BDL
3		Reverse	E6D-J2-B	BDL	BDL	BDL	BDL	BDL
3		Reverse	E6D-E7D-B	BDL	BDL	BDL	BDL	BDL
3		Reverse	E6D-J1-B	BDL	BDL	BDL	BDL	BDL
3		Reverse	E6D-E2-B	BDL	BDL	BDL	BDL	BDL
3		Reverse	E6D-K6-B	BDL	BDL	BDL	BDL	BDL
6		Forward	E9-pH4-B	BDL	BDL	BDL	BDL	BDL
1		Forward	E4D-048-B	0.16	BDL	BDL	BDL	BDL
1		Reverse	E4D-048-B	0.14	BDL	BDL	BDL	BDL
6		Reverse	E9-pH4-B	0.14	BDL	BDL	BDL	BDL
1		Forward	E4D-072-B	BDL	BDL	BDL	BDL	BDL
1		Reverse	E4D-072-B	BDL	BDL	BDL	BDL	BDL

Similarly, an acetonitrile wash was performed before and after each calibration standard (data not provided). The majority of the results were reported as BDL by the instrument. However, in a few cases the instrument reported a detection. These detections could be associated with instrument contamination, mainly as a result of contaminants building up on the analytical column. Isocratic HPLC analysis for explosives is especially prone to these types of false positives. This only appears to be an issue potentially affecting low-level detections, generally below the estimated reporting lim-

its. During the course of this project, the analytical column was changed three times.

Control results for each of the batch tests are presented in Table E-3. The control sample consisted of 70 mL DI spiked with the Restek standard to yield a concentration of 10 mg/L. The control concentration was consistently measured below 10 mg/L. As this issue was noted at the beginning of the project; considerable effort was spent investigating whether the standard received from Restek (10,000 mg/L standard) was at the designated concentration, or if it resulted from experimental error. The HPLC was calibrated with a number of different standards containing NG and/or DNTs and the Restek spiked standard was rerun (Table E-4). The first issue evaluated was whether there was an error in the dilution. The spiked standards were diluted by different individuals and had different standard concentrations. However, Table E-4 shows no difference in terms of accuracy between those samples that were run undiluted versus those that were diluted.

Also evaluated was whether the standards were at the specified concentration. It was determined that the calibration standard used for this project was slightly more than 1 mg/L. Consequently, all sample results are slightly under reported. However, since the same standard was utilized throughout the project, the relative difference between each of the batch tests remains constant. Therefore, the partitioning coefficient calculations remain unaffected.

A third issue assessed was whether the time lapsed from when the ampoule was opened had an effect on the measured concentration, presumably due to transformation processes (biodegradation or photo degradation). The Restek Project Standard of 4/15/08 was opened and immediately diluted and analyzed, thus minimizing the potential time for transformation to have occurred. However, the result of our analysis indicates this had no bearing on the measured concentration as it was still low relative to the manufacturer's reported concentration. We concluded that the cause of less-than-expected initial measured concentration in the controls is due to manufacturer variability in the supplied 10,000 mg/L standard. Because the actual measured concentration (or the average if there were multiple samples for a given test) was less than the intended spiked concentration provided by the manufacturer, the measured value was used as C_o in calculations for the batch tests.

Table E-3. Batch test control results (spiked concentration 10 mg/L) in order of analysis run.

Test Number	Vial Number	Forward/ Reverse	Sample #	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	1,2 Di NG (mg/L	1,3 Di NG (mg/L)
Pre-Test 2	A02	Forward	E3R-YB024-C	9.10	9.00	9.00	BDL*	BDL
Pre-Test 2	A05	Forward	E3R-YB000-C	9.50	9.50	9.70	BDL	BDL
Pre-Test 2	A06	Forward	E3R-NB000-C	10.0	10.2	10.2	BDL	BDL
Pre-Test 2	A08	Forward	E3R-YB024-C	9.00	8.90	9.10	BDL	BDL
Pre-Test 2	A08	Forward	E3R-YB024-C	9.00	9.10	9.00	BDL	BDL
Pre-Test 2	A06	Forward	E3R-NB000-C	10.0	10.0	10.2	BDL	BDL
Pre-Test 2	A05	Forward	E3R-YB000-C	9.50	9.50	9.30	BDL	BDL
Pre-Test 2	A02	Reverse	E3R-YB024-C	9.10	9.00	8.90	BDL	BDL
Pre-Test 2	A06	Reverse	E3R-NB000-C	10.0	10.2	10.2	BDL	BDL
Pre-Test 2	A06	Reverse	E3R-NB000-C	10.0	10.1	10.2	BDL	BDL
Pre-Test 2	A05	Reverse	E3R-YB000-C	9.54	9.49	9.74	BDL	BDL
Pre-Test 2	A05	Reverse	E3R-YB000-C	9.52	9.51	9.32	BDL	BDL
Pre-Test 2	A02	Reverse	E3R-YB024-C	9.07	9.03	9.04	BDL	BDL
Pre-Test 2	A08	Reverse	E3R-YB024-C	8.99	8.94	9.06	BDL	BDL
Pre-Test 2	A08	Reverse	E3R-YB024-C	9.02	9.05	8.99	BDL	BDL
Pre-Test 2	A02	Reverse	E3R-YB024-C	9.10	8.96	8.88	BDL	BDL
1	A24	Forward	E4-000-C	9.14	8.93	9.36	BDL	BDL
1	A29	Reverse	E4-000-C	9.07	8.96	9.23	BDL	BDL
1	A26	Forward	E4-024-C	9.16	8.94	9.13	BDL	BDL
1	A18	Forward	E4-048-C	9.12	9.17	9.33	BDL	BDL
1	A10	Forward	E4-072-C	8.97	8.98	9.03	BDL	BDL
1	A06	Forward	E4-120-C	9.18	9.09	9.23	BDL	BDL
1	A25	Forward	E4-216-C	9.14	8.98	9.39	BDL	BDL
5	A27	Forward	E8-T12-C	8.95	8.26	8.89	BDL	BDL
5	A22	Forward	E8-T32-C	8.90	8.25	8.90	BDL	BDL
5	A27	Reverse	E8-T12-C	8.96	8.25	8.82	BDL	BDL
5	A22	Reverse	E8-T32-C	8.94	8.39	8.96	BDL	BDL
4		Forward	E7-Control	9.08	8.28	9.05	BDL	BDL
4		Reverse	E7-Control	9.02	8.41	8.95	BDL	BDL
6		Reverse	E9-PH9-C	9.19	8.45	8.59	BDL	BDL
9		Forward	E12 -K7-NB-C-rain	9.16	9.16	8.93	BDL	BDL
9		Reverse	E12 -K7-NB-C-rain	9.24	9.08	9.19	BDL	BDL
9		Forward	E12 -K7-YB-C-rain	9.24	9.31	9.07	BDL	BDL
9		Reverse	E12 -K7-YB-C-rain	9.41	9.25	9.38	BDL	BDL
6		Forward	E9-pH4-C	8.60	9.18	9.14	BDL	BDL
6		Reverse	E9-pH4-C	8.47	9.15	9.07	BDL	BDL
			Mean	9.21	9.08	9.23	BDL	BDL
* BDL – Below MDL.								

Table E-4. Evaluation of different standards with different dilutions.

Order of Analysis	Standard Source	Date of Standard	Dilution Factor	Concentration (mg/L)	Measured Concentration (mg/L)				
					2,4-DNT	2,6-DNT	NG	1,2 Di NG	1,3 Di NG
Forward	Restek Project Standard	4/15/08	10000	1	0.928	0.945	0.916	BDL*	BDL
Reverse	Restek Project Standard (RPS)	4/15/08	10000	1	0.930	0.936	0.920	BDL	BDL
Forward	RPS	4/15/08	1000	1	0.930	0.951	0.938	BDL	BDL
Reverse	RPS	4/15/08	1000	1	0.919	0.939	0.936	BDL	BDL
Forward	SRB	Feb-08	10	1	1.03	1.04	1.04	BDL	BDL
Reverse	SRB	Feb-08	10	1	1.00	1.01	1.03	BDL	BDL
Forward	MEW	4/15/08	10	1	BDL	BDL	0.941	BDL	BDL
Reverse	MEW	4/15/08	10	1	BDL	BDL	0.968	BDL	BDL
Forward	SRB	4/16/08	10	1	BDL	BDL	0.942	BDL	BDL
Reverse	SRB	4/16/08	10	1	BDL	BDL	0.909	BDL	BDL
Forward	RPS	4/15/08	NA	10	9.16	9.31	9.17	BDL	BDL
Reverse	RPS	4/15/08	NA	10	9.11	9.30	9.06	BDL	BDL
Forward	RPS	4/15/08	NA	10	8.98	9.11	9.00	BDL	BDL
Reverse	RPS	4/15/08	NA	10	9.02	9.23	9.00	BDL	BDL
Forward	SRB	Feb-08	NA	10	10.1	10.2	10.1	BDL	BDL
Reverse	SRB	Feb-08	NA	10	9.89	9.94	9.97	BDL	BDL
Forward	RPS	4/15/08	1000	10	BDL	BDL	9.43	BDL	BDL
Reverse	RPS	4/15/08	1000	10	BDL	BDL	9.36	BDL	BDL
Forward	RPS	4/16/08	1000	10	BDL	BDL	9.27	BDL	0.135 J
Reverse	RPS	4/16/08	1000	10	BDL	BDL	9.26	BDL	0.135 J
BDL = Below MDL, RPS = Restek Project Standard									

All dilutions were performed with glass pipettes and Class A volumetric glassware, and within known tolerance measures. The same and different individuals performed dilutions and experiments at the start of the project multiple times.

As noted in Table E-4, the concentration measured was slightly less than the intended spiked concentration. This proved true even for Test 2 samples, which were spiked at varying concentrations (Table E-5). For this reason, the actual measured concentration or the average (in the case of multiple samples for a given test) was used as C_o for any calculations.

To evaluate experimental error associated with each batch test, all were conducted in triplicate. Figures E-1, E-2, and E-3 plot the Replicate 1 K_d

adsorption and desorption results for each experiment against its associated Replicate 2 and 3 K_d adsorption

Table E-5. Control results (spiked concentration varied) from Test 2 in order of analysis run.

Spiked Concentration (mg/L)	Vial #	Forward/Reverse	Sample #	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	1,2 GDN (mg/L)	1,3 GDN (mg/L)
0.1	A06	Forward	E5-010-C	0.094 J	0.10 J	0.084 J	BDL*	BDL
0.1	A06	Reverse order	E5-010-C	0.09 J	0.092 J	0.077 J	BDL	BDL
			Mean	0.09	0.10	0.08	BDL	BDL
1	A12	Forward	E5-100-C	0.90	0.92	0.902	BDL	BDL
1	A12	Reverse order	E5-100-C	0.89	0.91	0.9	BDL	BDL
			Mean	0.89	0.91	0.90	BDL	BDL
40	A10	Forward	E5-400-C	36.7	37.1	37.5	BDL	BDL
40	A18	Forward	E5-400-C	37.2	37.3	37.4	BDL	BDL
40	A10	Reverse order	E5-400-C	37.1	37.7	37.5	BDL	BDL
40	A18	Reverse order	E5-400-C	36.8	37.4	37.3	BDL	BDL
			Mean	37.0	37.4	37.4	BDL	BDL
80	A03	Forward	E5-800-C	72.5	73.4	74.1	BDL	BDL
80	A03	Reverse order	E5-800-C	73.3	72.9	74.3	BDL	BDL
			Mean	72.9	73.1	74.2	BDL	BDL
* BDL – Below MDL.								

and desorption results for batch Tests 1, 2, 3, 4, 5, 6, and 9. A legend is not included with the figures since all of the data is provided, and thus there are four associated symbols for each batch test. Figure E-1 shows the adsorption and desorption K_d results for NG, which indicates good replication for each experiment for all of the tests (the data plots along a straight line). The data plotted with negative values are associated with desorption experiments, principally in Replicate 3 for Test 5d.

A slightly greater variance in replication is evident for 2,4-DNT (Figure E-2). The negative K_d values in this figure are primarily associated with the replicate Test 5d results. The blue plus-sign outliers above the general trend of the data, as well as those below the general trend line around a K_d of 10 along the × -axis, are the Test 2d desorption Replicate 2 and 3d results. The outliers around a K_d of 35 L/kg along the × -axis are the desorption Test 3d results.

The 2,6-DNT results (Figure E-3) show less variation than the 2,4-DNT and NG data and indicate good replication of results. The data with the negative K_d values with pink symbols are the results from Test 4d, and the values with the blue symbols are from Test 2d.

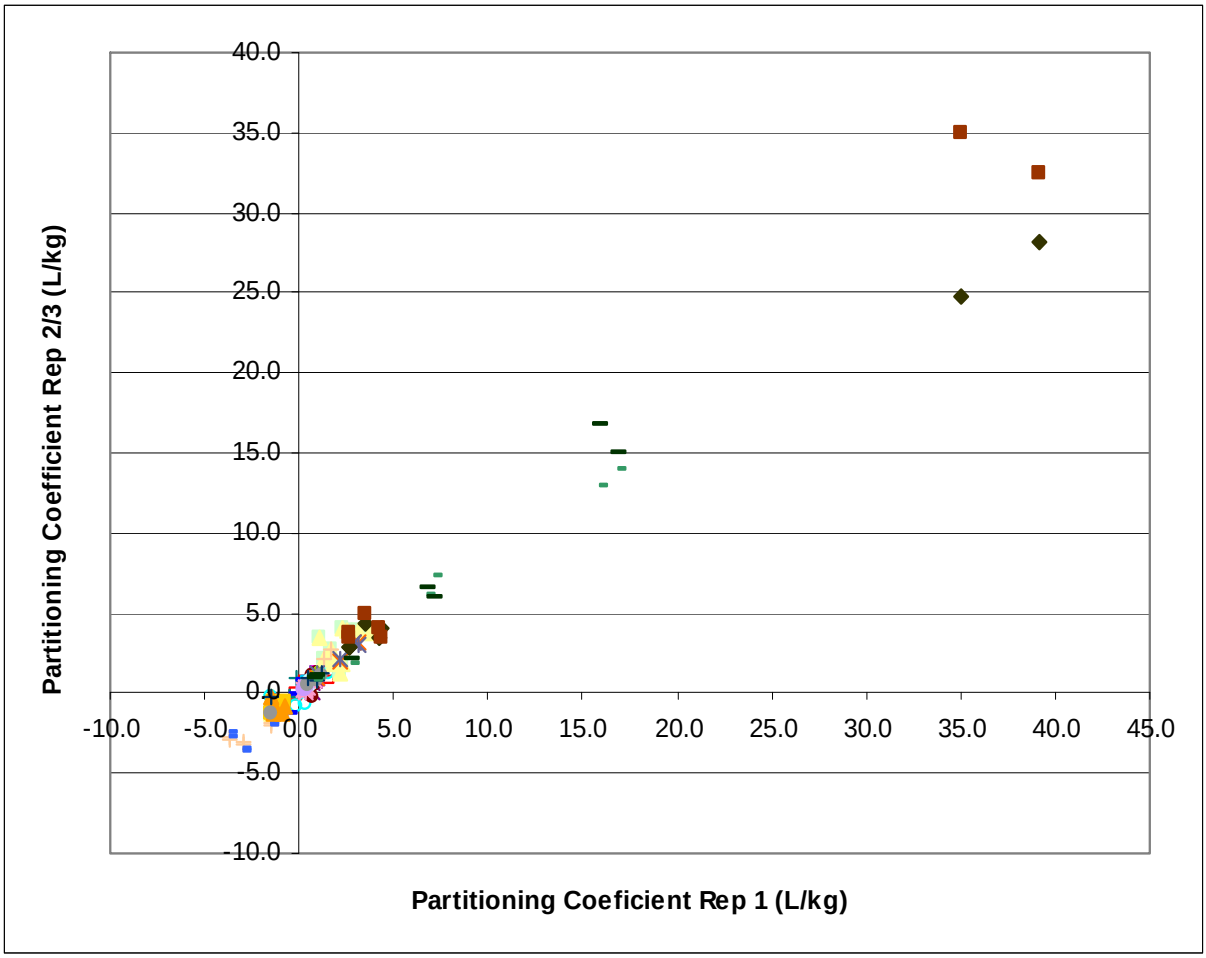

Figure E-1. Comparison of batch test replicate results for NG.

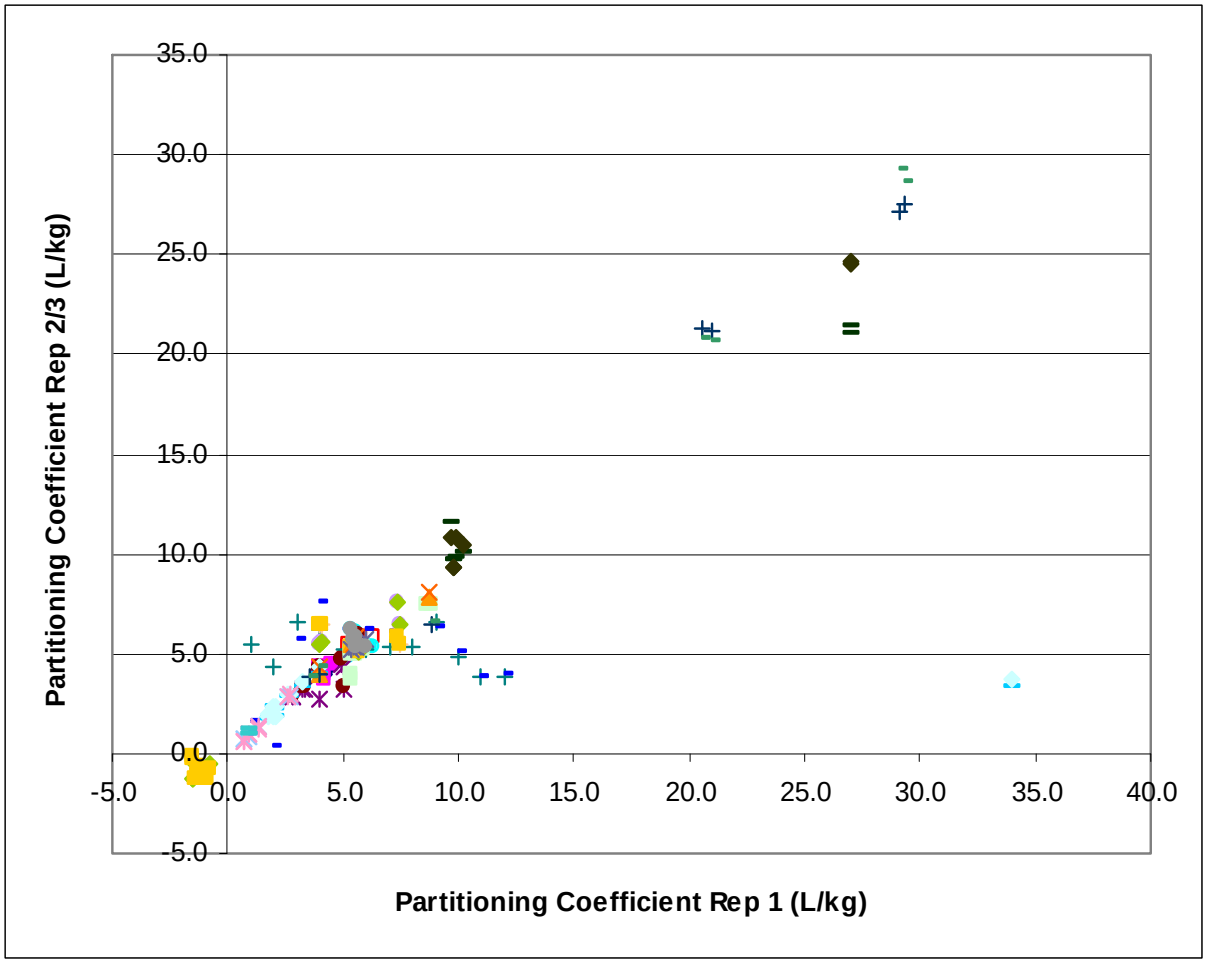

Figure E-2. Comparison of batch test replicate results for 2,4-DNT.

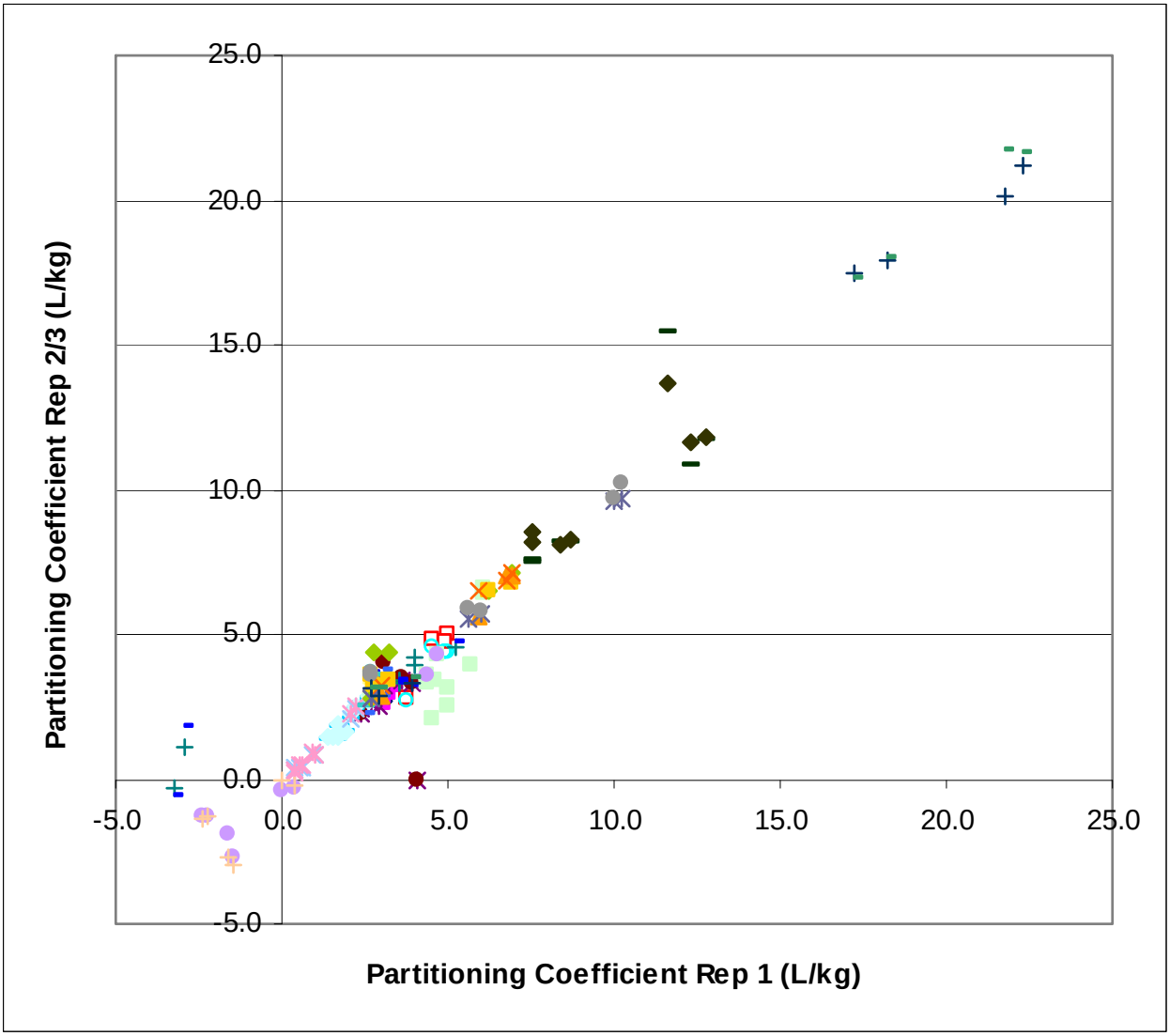

Figure E-3. Comparison of batch test replicate results for 2,6-DNT.

Appendix F

HPLC Batch Test Data Results

Test 1 - Equilibration Time								Test 1d - Equilibration Time							
		Sorption Aqueous Concentrations C_t (mg/L)								Desorption Aqueous Concentrations C_e (mg/L)					
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN		Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN	
0 hours	E4-000-1	5.08	5.64	7.69	BDL	BDL		0 hours	E4D-000-1	1.205	1.385	1.519	BDL	BDL	
	E4-000-2	4.31	4.94	7.32	BDL	BDL			E4D-000-2	0.924	1.028	1.133	BDL	BDL	
	E4-000-3	5.43	6.03	8.01	BDL	BDL			E4D-000-3	1.092	1.262	1.233	BDL	BDL	
	E4-000-B	BDL	0.07 J	BDL	BDL	BDL		R	E4D-000-1	1.157	1.380	1.534	BDL	BDL	
	E4-000-C	9.14	8.93	9.36	BDL	BDL		R	E4D-000-2	0.897	1.030	1.126	BDL	BDL	
dup	E4-000-C	9.07	8.96	9.23	BDL	BDL		R	E4D-000-3	1.058	1.252	1.227	BDL	BDL	
24 hours	E4-024-1	5.07	5.63	7.77	BDL	BDL		24 hours	E4D-024-1	2.113	2.412	2.726	0.293	BDL	
	E4-024-2	5.08	5.67	7.7	BDL	BDL			E4D-024-2	2.123	2.367	2.665	0.288	BDL	
	E4-024-3	4.9	5.51	7.51	BDL	BDL		dup	E4D-024-2	2.125	2.373	2.676	0.274	BDL	
	E4-024-B	BDL	BDL	BDL	BDL	BDL			E4D-024-3	2.017	2.205	2.283	0.256	BDL	
	E4-024-C	9.16	8.94	9.13	BDL	BDL		R	E4D-024-1	2.056	2.398	2.726	0.287	BDL	
								R	E4D-024-2	2.072	2.362	2.669	0.284	BDL	
48 hours	E4-048-1	4.97	5.54	7.6	BDL	BDL		R dup	E4D-024-2	2.072	2.357	2.675	0.280	BDL	
dup	E4-048-1	5.13	5.72	7.82	BDL	BDL		R	E4D-024-3	2.032	2.209	2.292	0.245 J	BDL	
	E4-048-2	5.09	5.83	7.55	BDL	BDL									
	E4-048-3	5.23	5.95	7.9	BDL	BDL		48 hours	E4D-048-1	1.933	2.052	1.752	0.247 J	BDL	
	E4-048-B	0.07 J	BDL	BDL	BDL	BDL			E4D-048-2	2.070	2.243	1.893	0.249 J	BDL	
	E4-048-C	9.12	9.17	9.33	BDL	BDL			E4D-048-3	2.017	2.205	2.116	0.262	BDL	
									E4D-048-B	BDL	BDL	BDL	BDL	BDL	
72 hours	E4-072-1	4.95	5.51	7.58	0.38	BDL		R	E4D-048-1	1.889	2.041	1.741	0.261	0.216 J	
	E4-072-2	4.95	5.48	7.58	BDL	BDL		R	E4D-048-2	2.031	2.234	1.912	0.250 J	0.218 J	
	E4-072-3	5.09	5.63	7.68	BDL	BDL		R	E4D-048-3	1.974	2.206	2.115	0.280	0.189 J	
	E4-072-B	BDL	0.07 J	BDL	BDL	BDL		R	E4D-048-B	BDL	BDL	BDL	BDL	BDL	
	E4-072-C	8.97	8.98	9.03	BDL	BDL									
								72 hours	E4D-072-1	1.982	1.907	1.600	0.230 J	BDL	
120 hours	E4-120-1	5.01	5.59	7.56	BDL	BDL			E4D-072-2	2.047	1.932	1.768	0.273	BDL	
	E4-120-2	4.96	5.61	7.66	BDL	BDL			E4D-072-3	2.105	1.973	1.585	0.236 J	BDL	
	E4-120-3	5.03	5.68	7.89	BDL	BDL			E4D-072-B	BDL	BDL	BDL	BDL	BDL	
	E4-120-B	0.05 J	BDL	BDL	BDL	BDL		R	E4D-072-1	1.966	1.889	1.589	0.216 J	BDL	
	E4-120-C	9.18	9.09	9.23	BDL	BDL		R	E4D-072-2	2.034	1.904	1.738	0.218 J	BDL	
								R	E4D-072-3	2.085	1.940	1.560	0.189 J	BDL	
216 hours	E4-216-1	4.78	5.53	7.41	0.65	BDL		R	E4D-072-B	BDL	BDL	BDL	BDL	BDL	
dup	E4-216-1	4.73	5.36	7.28	0.62	BDL									
	E4-216-2	4.81	5.44	7.32	BDL	BDL		120 hours	E4D-120-1	2.151	1.887	2.165	0.259	BDL	
	E4-216-3	4.81	5.47	7.39	0.63	BDL			E4D-120-2	2.129	1.920	2.073	0.258	BDL	
	E4-216-B	0.05 J	0.16 J	BDL	BDL	BDL			E4D-120-3	2.193	1.954	2.199	0.293	BDL	
	E4-216-C	9.14	8.98	9.39	BDL	BDL		R	E4D-120-1	2.156	1.921	2.144	0.296	BDL	
								R	E4D-120-2	2.122	1.939	2.038	0.309	BDL	
	ACN Wash	BDL	BDL	BDL	BDL	BDL		R	E4D-120-3	2.210	1.982	2.177	0.335	BDL	
	ACN Wash	BDL	BDL	BDL	BDL	BDL			E4D-120-B	BDL	BDL	BDL	BDL	BDL	
	ACN Wash	BDL	BDL	BDL	BDL	BDL		R	E4D-120-B	BDL	BDL	BDL	BDL	BDL	
	ACN Wash	BDL	BDL	BDL	BDL	BDL									
	ACN Wash	BDL	BDL	BDL	BDL	BDL		240 hours	E4D-240-1	2.086	2.009	1.739	0.346	BDL	
	ACN Wash	BDL	BDL	BDL	BDL	BDL			E4D-240-2	2.134	2.030	1.881	0.281	BDL	
	ACN Wash	BDL	BDL	BDL	BDL	BDL			E4D-240-3	2.056	2.007	1.659	0.311	BDL	
	ACN Wash	BDL	BDL	BDL	BDL	BDL									
	Calibration	10	10	10	10	10			ACN Wash	BDL	BDL	BDL	BDL	BDL	
	Calibration	10	10	10	10	10			Calibration	1.000	1.000	1.000	1.000	1.000	
	Calibration	10	10	10	10	10			ACN Wash	BDL	BDL	BDL	BDL	BDL	
	Cal-Unknown	10.12	9.91	10.22	10.04	10									

HPLC Batch Test Data (cont.)

Test 2 - NG/DNT Concentration												Test 2d - NG/DNT Concentration											
		Sorption Aqueous Concentrations C_t (mg/L)												Desorption Aqueous Concentrations C_e (mg/L)									
Other	Sample	2,4-DNT		2,6-DNT		NG		1,2-GDN		1,3-GDN		Other	Sample	2,4-DNT		2,6-DNT		NG		1,2-GDN		1,3-GDN	
0.1 ppm	E5-010-1	0.043	J	0.059	J	0.071	J	BDL		BDL			E5D-010-1	0.065	J	0.108	J	0.076	J	BDL		BDL	
dup	E5-010-1	0.042	J	0.052	J	0.076	J	0.056	J	BDL			E5D-010-2	0.024	J	BDL		BDL		BDL		BDL	
	E5-010-2	0.044	J	0.058	J	0.079	J	0.090	J	BDL			E5D-010-3	0.040	J	BDL		BDL		BDL		BDL	
	E5-010-3	0.042	J	0.053	J	0.083	J	BDL		BDL													
	E5-010-C	0.094	J	0.100	J	0.084	J	BDL		BDL													
1.0 ppm	E5-100-1	0.449		0.528		0.757		0.086	J	BDL			E5D-100-1	0.207		0.231		0.211	J	BDL		BDL	
	E5-100-2	0.468		0.543		0.768		0.084	J	BDL			E5D-100-2	0.196		0.218		0.190	J	BDL		BDL	
	E5-100-3	0.461		0.538		0.764		0.088	J	BDL			E5D-100-3	0.203		0.228		0.185	J	BDL		BDL	
	E5-100-C	0.897		0.919		0.902		BDL		BDL													
40.0 ppm	E5-400-1	22.38		24.2		31.4		0.29		BDL			E5D-400-1	8.212		7.309		7.966		0.557		0.106	J
	E5-400-2	22.47		24.46		31.33		0.26		BDL			E5D-400-2	7.944		7.568		6.420		0.143	J	BDL	
	E5-400-3	21.96		24.09		31.58		BDL		BDL			E5D-400-3	8.026		7.685		6.548		0.140	J	BDL	
	E5-400-C	36.72		37.07		37.52		BDL		BDL													
dup	E5-400-C	37.24		37.32		37.41		BDL		BDL													
80.0 ppm	E5-800-1	46.75		49.7		64.06		0.51		0.12	J		E5D-800-1	18.452		15.489		20.372		0.978		0.261	
	E5-800-2	46.49		49.71		63.52		0.48		0.11	J		E5D-800-2	16.613		15.657		13.513		0.254		0.065	J
	E5-800-3	46.63		49.79		63.08		0.52		0.1	J		E5D-800-3	16.518		15.759		13.789		0.245	J	0.069	J
	E5-800-C	72.46		73.41		74.08		BDL		BDL													
	E5-Blank	BDL		BDL		BDL		BDL		BDL													
	ACN Wash	BDL		BDL		BDL		BDL		BDL													
	ACN Wash	BDL		BDL		BDL		BDL		BDL													
	ACN Wash	BDL		BDL		BDL		BDL		BDL													
	ACN Wash	BDL		BDL		BDL		BDL		BDL													
	Calibration	1.000		1.000		1.000		1.000		1.000													
	Calibration	1.000		1.000		1.000		1.000		1.000													
R	E5-010-1	0.051	J	0.044	J	0.071	J	BDL		BDL		R	E5D-010-1	0.074	J	0.106	J	0.065	J	BDL		BDL	
R dup	E5-010-1	0.046	J	0.060	J	0.070	J	BDL		BDL		R	E5D-010-2	0.033	J	0.041	J	BDL		BDL		BDL	
R	E5-010-2	0.060	J	0.063	J	0.072	J	BDL		BDL		R	E5D-010-3	0.024	J	0.042	J	BDL		BDL		BDL	
R	E5-010-3	0.050	J	0.059	J	0.066	J	0.092	J	BDL													
R	E5-010-C	0.090	J	0.092	J	0.077	J	BDL		BDL													
R	E5-100-1	0.448		0.510		0.779		BDL		BDL		R	E5D-100-1	0.221		0.240		0.211	J	BDL		BDL	
R	E5-100-2	0.473		0.550		0.775		0.061	J	BDL		R	E5D-100-2	0.200		0.222		0.200	J	BDL		BDL	
R	E5-100-3	0.455		0.550		0.816		BDL		BDL		R	E5D-100-3	0.185		0.231		0.187	J	BDL		BDL	
R	E5-100-C	0.890		0.905		0.900		BDL		BDL													
R	E5-400-1	22.44		23.95		31.37		BDL		0.06	J	R	E5D-400-1	8.226		7.278		8.070		0.544		0.116	J
R	E5-400-2	22.64		24.1		31.37		0.28		BDL		R	E5D-400-2	8.012		7.637		6.558		0.134	J	BDL	
R	E5-400-3	22.01		23.81		31.56		0.29		BDL		R	E5D-400-3	8.062		7.706		6.671		0.123	J	BDL	
R	E5-400-C	37.05		37.69		37.54		BDL		BDL													
R dup	E5-400-C	36.81		37.35		37.28		BDL		BDL													
R	E5-800-1	46.65		49.64		63.45		0.47		0.09	J	R	E5D-800-1	18.397		15.523		20.654		0.966		0.261	
R	E5-800-2	46.28		49.99		63.37		0.51		0.15	J	R	E5D-800-2	16.641		15.652		13.667		0.252		0.071	J
R	E5-800-3	46.21		49.8		62.76		0.47		BDL		R	E5D-800-3	16.389		15.676		13.911		0.231	J	0.066	J
R	E5-800-C	73.33		72.88		74.27		BDL		BDL													
R	E5-Blank	BDL		0.16	J	0.12	J	BDL		BDL													
R																							
R	ACN Wash	BDL		BDL		BDL		BDL		BDL													
R	ACN Wash	BDL		BDL		BDL		BDL		BDL													
R	ACN Wash	BDL		BDL		BDL		BDL		BDL													
R	ACN Wash	BDL		BDL		BDL		BDL		BDL													
R	ACN Wash	BDL		BDL		BDL		BDL		BDL													
R	ACN Wash	BDL		BDL		BDL		BDL		BDL													
R	Cal-Unknown	1.002		0.992		0.988		0.995		1.006													
R	Calibration	1.000		1.000		1.000		1.000		1.000													
R	Calibration	1.000		1.000		1.000		1.000		1.000													

HPLC Batch Test Data (cont.)

Test 3 - Surface Soil Heterogeneity								Test 3d - Surface Soil Heterogeneity							
		Sorption Aqueous Concentrations C_t (mg/L)								Desorption Aqueous Concentrations C_e (mg/L)					
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN		1,3-GDN	Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN		1,3-GDN
Echo range #1	E6-E1-1	6.56	6.82	8.13	0.25	J	BDL		E6D-E1-1	1.440	1.369	0.935	0.122	J	BDL
R	E6-E1-1	6.7	7.03	8.23	BDL		BDL	R	E6D-E1-1	1.445	1.368	0.937	0.115	J	BDL
	E6-E1-2	6.8	7.04	8.2	BDL		BDL		E6D-E1-2	1.460	1.567	0.924	0.134	J	BDL
R	E6-E1-2	6.81	7.3	8.16	BDL		BDL	R	E6D-E1-2	1.484	1.574	0.941	0.133	J	BDL
	E6-E1-3	6.75	7.11	8.26	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
dup	E6-E1-3	6.8	7.07	8.17	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
R	E6-E1-3	6.74	7.16	8.15	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
R dup	E6-E1-3	6.81	7.33	8.03	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
Echo range #2	E6-E2-1	6.87	7.17	8.35	BDL		BDL		E6D-E2-1	1.391	1.302	0.976	0.056	J	BDL
R	E6-E2-1	6.87	7.31	8.37	BDL		BDL	R	E6D-E2-1	1.386	1.303	0.979	0.069	J	BDL
	E6-E2-2	6.92	7.17	8.28	BDL		BDL		E6D-E2-2	1.388	1.508	0.818	0.101	J	BDL
R	E6-E2-2	7	7.3	8.36	BDL		BDL	R	E6D-E2-2	1.385	1.505	0.817	0.102	J	BDL
	E6-E2-3	6.75	7.21	8.24	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
R	E6-E2-3	6.64	7.22	8.05	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
Juliet range #1	E6-J1-1	6.58	6.99	8.27	BDL		BDL		E6D-J1-1	1.472	1.471	1.093	0.102	J	BDL
R	E6-J1-1	6.65	6.9	8.39	BDL		BDL	R	E6D-J1-1	1.487	1.486	1.131	0.099	J	BDL
	E6-J1-2	6.46	6.9	8.23	BDL		BDL		E6D-J1-2	1.582	1.674	1.049	0.171	J	BDL
dup	E6-J1-2	6.4	6.91	7.93	BDL		BDL	R	E6D-J1-2	1.595	1.662	1.029	0.161	J	BDL
R	E6-J1-2	6.49	6.83	8.19	BDL		BDL								
R dup	E6-J1-2	6.43	7	8.13	BDL		BDL								
	E6-J1-3	6.39	6.75	8.15	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
R	E6-J1-3	6.34	6.89	8.26	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
Juliet range #2	E6-J2-1	5.65	6.18	7.69	0.26		BDL		E6D-J2-1	1.466	1.592	1.355	0.118	J	BDL
R	E6-J2-1	5.57	6.16	7.72	BDL		BDL	R	E6D-J2-1	1.476	1.599	1.371	0.111	J	BDL
	E6-J2-2	5.53	5.99	7.82	BDL		BDL		E6D-J2-2	1.813	1.776	1.381	0.158	J	BDL
R	E6-J2-2	5.53	6.06	7.76	BDL		BDL	R	E6D-J2-2	1.819	1.781	1.396	0.159	J	BDL
	E6-J2-3	5.43	5.92	7.72	0.26		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
R	E6-J2-3	5.38	6.06	7.63	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
Kilo range,west	E6-K6-1	6.11	6.48	7.91	BDL		BDL		E6D-K6-1	1.654	1.667	1.371	0.072	J	BDL
R	E6-K6-1	6.05	6.59	8	BDL		BDL	R	E6D-K6-1	1.656	1.674	1.386	0.075	J	BDL
	E6-K6-2	5.99	6.34	7.75	BDL		BDL		E6D-K6-2	1.841	1.898	1.411	0.077	J	BDL
R	E6-K6-2	5.93	6.59	7.95	BDL		BDL	R	E6D-K6-2	1.877	1.923	1.416	0.057	J	BDL
	E6-K6-3	5.87	6.25	7.92	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						
R	E6-K6-3	5.84	6.42	7.94	BDL		BDL		Rep 3 Soils Sacrificed for Soil Analysis						

HPLC Batch Test Data (cont.)

Test 4 - Depth Evaluation								Test 4d - Depth Evaluation							
		Sorption Aqueous Concentrations C_t (mg/L)								Desorption Aqueous Concentrations C_e (mg/L)					
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN		Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN		1,3-GDN
Kilo range 9-12"	E7-K3-1	7.34	7.93	8.5	BDL	BDL			E7D-K3-1	1.160	1.961	1.961	0.419		BDL
R	E7-K3-1	7.35	7.73	8.51	BDL	BDL		R	E7D-K3-1	1.191	1.970	1.939	0.395		BDL
	E7-K3-2	7.39	8.07	8.77	BDL	BDL			E7D-K3-2	1.165	1.886	1.715	0.360		BDL
R	E7-K3-2	7.35	8.04	8.67	BDL	BDL		R	E7D-K3-2	1.166	1.880	1.717	0.400		BDL
	E7-K3-3	7.42	7.92	8.7	BDL	BDL			E7D-K3-3	1.182	1.976	1.913	0.331		BDL
R	E7-K3-3	7.56	7.99	8.64	BDL	BDL		R	E7D-K3-3	1.193	1.960	1.886	0.398		BDL
Kilo range 18-24"	E7-K4-1	7.87	8.41	8.83	BDL	BDL			E7D-K4-1	0.836	1.825	2.127	0.341		BDL
dup	E7-K4-1	7.88	8.47	8.8	BDL	BDL		R	E7D-K4-1	0.847	1.834	2.126	0.378		BDL
R	E7-K4-1	7.96	8.55	8.98	BDL	BDL			E7D-K4-2	0.899	1.857	2.149	0.278		BDL
R dup	E7-K4-1	7.98	8.5	8.84	BDL	BDL		R	E7D-K4-2	0.915	1.866	2.123	0.291		BDL
	E7-K4-2	7.98	8.59	8.91	BDL	BDL			E7D-K4-3	0.890	1.906	2.172	0.356		BDL
R	E7-K4-2	7.97	8.58	9.02	BDL	BDL		R	E7D-K4-3	0.882	1.900	2.150	0.330		BDL
	E7-K4-3	7.79	8.5	8.91	BDL	BDL									
R	E7-K4-3	7.82	8.56	8.77	BDL	BDL									
Kilo range 30-36"	E7-K5-1	8.21	8.68	8.83	BDL	BDL			E7D-K5-1	0.609	1.599	2.076	0.140	J	BDL
R	E7-K5-1	8.18	8.62	8.84	BDL	BDL		R	E7D-K5-1	0.616	1.611	2.090	0.127	J	BDL
	E7-K5-2	8.12	8.78	8.8	BDL	BDL			E7D-K5-2	0.606	1.625	2.086	0.136	J	BDL
R	E7-K5-2	8.22	8.68	8.88	BDL	BDL		R	E7D-K5-2	0.623	1.626	2.058	0.118	J	BDL
	E7-K5-3	8.26	8.85	8.89	BDL	BDL			E7D-K5-3	0.586	1.595	2.109	0.123	J	BDL
R	E7-K5-3	8.32	8.94	8.95	BDL	BDL		R	E7D-K5-3	0.578	1.595	2.099	0.130	J	BDL
Echo range 9-12"	E7-E3-1	7.16	7	8.64	BDL	BDL			E7D-E3-1	1.097	0.864	0.500	0.088	J	BDL
R	E7-E3-1	7.14	6.95	8.7	BDL	BDL		R	E7D-E3-1	1.092	0.852	0.494	0.088	J	BDL
	E7-E3-2	7.26	7.15	8.79	BDL	BDL			E7D-E3-2	1.058	0.824	0.477	0.081	J	BDL
R	E7-E3-2	7.28	7.21	8.33	BDL	BDL		R	E7D-E3-2	1.058	0.831	0.513	0.085	J	BDL
	E7-E3-3	7.22	7.06	8.65	BDL	BDL			E7D-E3-3	1.070	0.852	0.490	0.090	J	BDL
R	E7-E3-3	7.27	7.14	8.63	BDL	BDL		R	E7D-E3-3	1.066	0.835	0.496	0.092	J	BDL
	E7-E3-B	BDL	BDL	BDL	BDL	BDL			E7D-E3-B	BDL	BDL	BDL	BDL		BDL
R	E7-E3-B	BDL	BDL	BDL	BDL	BDL		R	E7D-E3-B	BDL	BDL	BDL	BDL		BDL
Echo range 18-24"	E7-E4-1	8.01	7.5	8.47	BDL	BDL			E7D-E4-1	0.814	0.597	0.398	0.071	J	BDL
R	E7-E4-1	8.02	7.57	8.53	BDL	BDL		R	E7D-E4-1	0.808	0.599	0.411	0.061	J	BDL
	E7-E4-2	8.16	7.54	9.34	BDL	BDL			E7D-E4-2	0.785	0.613	0.359	0.063	J	BDL
R	E7-E4-2	8.05	7.6	8.87	BDL	BDL		R	E7D-E4-2	0.790	0.594	0.371	0.061	J	BDL
	E7-E4-3	8.19	7.56	8.9	BDL	BDL			E7D-E4-3	0.780	0.617	0.345	0.052	J	BDL
R	E7-E4-3	8.15	7.76	8.62	BDL	BDL		R	E7D-E4-3	0.779	0.606	0.355	0.061	J	BDL
	E7-E4-B	BDL	BDL	BDL	BDL	BDL			E7D-E4-B	BDL	BDL	BDL	BDL		BDL
R	E7-E4-B	BDL	BDL	BDL	BDL	BDL		R	E7D-E4-B	BDL	BDL	BDL	BDL		BDL
Echo range 30-36	E7-E5-1	7.94	7.59	9.02	BDL	BDL			E7D-E5-1	0.823	0.634	0.418	0.062	J	BDL
R	E7-E5-1	7.76	7.63	8.62	BDL	BDL		R	E7D-E5-1	0.818	0.625	0.405	0.057	J	BDL
	E7-E5-2	7.67	7.35	8.69	BDL	BDL			E7D-E5-2	0.814	0.615	0.410	0.061	J	BDL
R	E7-E5-2	7.63	7.45	8.59	BDL	BDL		R	E7D-E5-2	0.806	0.628	0.405	0.058	J	BDL
	E7-E5-3	7.64	7.36	8.93	BDL	BDL			E7D-E5-3	0.880	0.691	0.465	0.063	J	BDL
R	E7-E5-3	7.45	7.18	8.79	BDL	BDL		R	E7D-E5-3	0.882	0.689	0.450	0.069	J	BDL
	E7-E5-B	BDL	BDL	BDL	BDL	BDL			E7D-E5-B	BDL	BDL	BDL	BDL		BDL
R	E7-E5-B	BDL	BDL	BDL	BDL	BDL		R	E7D-E5-B	BDL	BDL	BDL	BDL		BDL
Juliet range 9-12"	E7-J3-1	7.9	7.41	8.96	BDL	BDL			E7D-J3-1	1.065	0.733	0.615	0.100	J	BDL
R	E7-J3-1	7.81	7.79	8.48	BDL	BDL		R	E7D-J3-1	1.048	0.719	0.591	0.106	J	BDL
	E7-J3-2	7.74	7.37	8.87	BDL	BDL			E7D-J3-2	1.033	0.709	0.499	0.089	J	BDL
R	E7-J3-2	7.87	7.52	8.67	BDL	BDL		R	E7D-J3-2	1.042	0.730	0.507	0.093	J	BDL
	E7-J3-3	7.65	7.33	8.77	BDL	BDL			E7D-J3-3	1.112	0.738	0.593	0.098	J	BDL
R	E7-J3-3	7.58	7.37	8.65	BDL	BDL		R	E7D-J3-3	1.100	0.743	0.564	0.104	J	BDL
	E7-J3-B	BDL	BDL	BDL	BDL	BDL			E7D-J3-B	BDL	BDL	BDL	BDL		BDL
R	E7-J3-B	BDL	BDL	BDL	BDL	BDL		R	E7D-J3-B	BDL	BDL	BDL	BDL		BDL
Juliet range 18-24"	E7-J4-1	7.99	8.05	8.93	BDL	BDL			E7D-J4-1	0.929	0.441	0.519	0.059	J	BDL
R	E7-J4-1	8.07	7.96	9.21	BDL	BDL		R	E7D-J4-1	0.938	0.436	0.515	0.059	J	BDL
	E7-J4-2	8.08	7.94	9.01	BDL	BDL			E7D-J4-2	0.939	0.421	0.536	0.066	J	BDL
R	E7-J4-2	8.04	7.97	9.01	BDL	BDL		R	E7D-J4-2	0.929	0.432	0.525	0.068	J	BDL
	E7-J4-3	8.02	7.97	9.2	BDL	BDL			E7D-J4-3	0.954	0.467	0.514	0.069	J	BDL
R	E7-J4-3	8.03	7.96	8.93	BDL	BDL		R	E7D-J4-3	0.947	0.456	0.515	0.071	J	BDL
	E7-J4-B	BDL	BDL	BDL	BDL	BDL			E7D-J4-B	BDL	BDL	BDL	BDL		BDL
	E7-J4-B	BDL	BDL	0.22	J BDL	BDL		R	E7D-J4-B	BDL	BDL	BDL	BDL		BDL

HPLC Batch Test Data (Cont.)

Test 4 (Cont.)

Test 4 - Depth Evaluation (cont.)								Test 4d - Depth Evaluation (cont.)						
		Sorption Aqueous Concentrations C_t (mg/L)								Desorption Aqueous Concentrations C_e (mg/L)				
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN		Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN
Juliet range 30-36"	E7-J5-1	8.75	8.04	8.9	BDL	BDL			E7D-J5-1	0.479	0.255	0.351	BDL	BDL
R	E7-J5-1	8.78	8.22	8.91	BDL	BDL		R	E7D-J5-1	0.463	0.234	0.365	BDL	BDL
	E7-J5-2 repeat	8.49	8.04	9.00	BDL	BDL			E7D-J5-2 repeat	0.571	0.285	0.569	BDL	BDL
R	E7-J5-2 repeat	8.80	8.10	9.22	BDL	BDL		R	E7D-J5-2 repeat	0.564	0.272	0.585	BDL	BDL
R dup	E7-J5-2 repeat	8.67	8.09	9.17	BDL	BDL			E7D-J5-3 repeat	0.487	0.240	0.413	BDL	BDL
	E7-J5-3 repeat	8.76	8.89	9.10	BDL	BDL		R	E7D-J5-3 repeat	0.487	0.242	0.414	BDL	BDL
R	E7-J5-3 repeat	8.58	7.97	8.86	BDL	BDL			E7D-J5-B	BDL	BDL	BDL	BDL	BDL
	E7-J5-B	BDL	BDL	BDL	BDL	BDL		R	E7D-J5-B	BDL	BDL	BDL	BDL	BDL
R	E7-J5-B	BDL	BDL	BDL	BDL	BDL								
	E7-Control	9.08	8.28	9.05	BDL	BDL			Calibration	1.000	1.000	1.000	1.000	1.000
	E7-Control	9.02	8.41	8.95	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	Calibration	1.000	1.000	1.000	1.000	1.000								
	Calibration	1.000	1.000	1.000	1.000	1.000								

Test 5 - Temperature Evaluation								Test 5d - Temperature Evaluation							
		Sorption Aqueous Concentrations C_t (mg/L)								Desorption Aqueous Concentrations C_e (mg/L)					
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN		Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN		1,3-GDN
12ºC	E8-T12-1	4.92	5.1	7.53	BDL	BDL			E8D-T12-1	1.735	1.470	2.129	0.230	J	BDL
dup	E8-T12-1	4.89	5.05	7.48	BDL	BDL			E8D-T12-2	1.901	1.609	2.133	0.223	J	BDL
	E8-T12-2	4.44	4.68	7.42	BDL	BDL			E8D-T12-3	1.898	1.598	2.327	0.233	J	BDL
	E8-T12-3	4.52	4.79	7.35	BDL	BDL									
	E8-T12-B	BDL	BDL	BDL	BDL	BDL									
	E8-T12-C	8.95	8.26	8.89	BDL	BDL									
									E8D-T32-1	2.290	1.886	2.468	0.400		BDL
32ºC	E8-T32-1	5.37	5.52	7.53	BDL	BDL			E8D-T32-2	1.934	1.568	2.088	0.347		BDL
	E8-T32-2	5.44	5.66	7.56	BDL	BDL			E8D-T32-3	2.157	1.824	2.272	0.368		BDL
	E8-T32-3	5.25	5.56	7.38	BDL	BDL									
	E8-T32-B	BDL	BDL	BDL	BDL	BDL									
	E8-T32-C	8.9	8.25	8.9	BDL	BDL									
	ACN Wash	BDL	BDL	BDL	BDL	BDL									
	ACN Wash	BDL	BDL	BDL	BDL	BDL									
	Calibration	1.000	1.000	1.000	1.000	1.000									
R	E8-T12-1	4.98	5.21	7.53	BDL	BDL									
R dup	E8-T12-1	4.92	5.21	7.56	BDL	BDL		R	E8D-T12-1	1.747	1.505	2.208	0.231	J	BDL
R	E8-T12-2	4.48	4.8	7.43	BDL	BDL		R	E8D-T12-2	1.894	1.634	2.187	0.216	J	BDL
R	E8-T12-3	4.52	4.8	7.49	BDL	BDL		R	E8D-T12-3	1.898	1.633	2.395	0.227	J	BDL
R	E8-T12-B	BDL	BDL	BDL	BDL	BDL									
R	E8-T12-C	8.96	8.25	8.82	BDL	BDL									
R	E8-T32-1	5.37	5.68	7.54	BDL	BDL		R	E8D-T32-1	2.278	1.894	2.489	0.380		BDL
R	E8-T32-2	5.41	5.64	7.36	BDL	BDL		R	E8D-T32-2	1.942	1.592	2.160	0.350		BDL
R	E8-T32-3	5.27	5.59	7.57	BDL	BDL		R	E8D-T32-3	2.150	1.842	2.341	0.352		BDL
R	E8-T32-B	BDL	BDL	BDL	BDL	BDL									
R	E8-T32-C	8.94	8.39	8.96	BDL	BDL									
	ACN Wash	BDL	BDL	BDL	BDL	BDL									
	ACN Wash	BDL	BDL	BDL	BDL	BDL									
	ACN Wash	BDL	BDL	BDL	BDL	BDL									
	ACN Wash	BDL	BDL	BDL	BDL	BDL									
	Calibration	1.000	1.000	1.000	1.000	1.000									
	Cal-Unknown	1.012	1.005	1.007	1.003	1.006									

HPLC Batch Test Data (cont.)

Test 6 - pH							Test 6d - pH						
		Sorption Aqueous Concentrations C_t (mg/L)							Desorption Aqueous Concentrations C_e (mg/L)				
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN	Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN
pH=4.2	E9-pH4-1	3.87	4.07	5.79	BDL	BDL		E9D-PH4-1	2.225	1.699	3.005	0.169 J	BDL
Dup	E9-pH4-1	3.84	4.04	5.59	BDL	BDL	R	E9D-PH4-1	2.204	1.673	2.922	0.168 J	BDL
R	E9-pH4-1	3.86	4.09	5.64	BDL	BDL		E9D-PH4-2	2.288	1.753	3.171	0.189 J	BDL
R dup	E9-pH4-1	3.8	3.97	5.51	BDL	BDL	R	E9D-PH4-2	2.274	1.740	3.110	BDL	BDL
	E9-pH4-2	3.83	4.05	5.61	BDL	BDL		E9D-PH4-3	2.189	1.739	2.871	BDL	BDL
R	E9-pH4-2	3.82	4.05	5.58	BDL	BDL	R	E9D-PH4-3	2.178	1.717	2.805	0.178 J	BDL
	E9-pH4-3	3.86	4.06	5.65	BDL	BDL		E9D-PH4-B	BDL	BDL	BDL	BDL	BDL
R	E9-pH4-3	3.78	3.93	5.66	BDL	BDL	R	E9D-PH4-B	BDL	BDL	BDL	BDL	BDL
	E9-pH4-B	BDL	BDL	BDL	BDL	BDL							
R	E9-pH4-B	BDL	0.14 J	BDL	BDL	BDL							
	E9-pH4-C	9.18	8.6	9.14	BDL	BDL							
R	E9-pH4-C	9.15	8.47	9.07	BDL	BDL							
pH=8.2	E9-PH9-1	3.374	3.937	6.151	BDL	BDL		E9D-pH9-1	2.389	2.476	2.520	BDL	BDL
R	E9-PH9-1	3.303	3.668	5.981	BDL	BDL	R	E9D-pH9-1	2.381	2.488	2.509	BDL	BDL
	E9-PH9-2	3.625	4.056	6.319	BDL	BDL		E9D-pH9-2	2.350	2.423	2.422	BDL	BDL
R	E9-PH9-2	3.520	4.013	6.148	BDL	BDL	R	E9D-pH9-2	2.344	2.414	2.429	BDL	BDL
	E9-PH9-3	3.526	3.754	6.192	BDL	BDL		E9D-pH9-3	2.438	2.489	2.511	BDL	BDL
R	E9-PH9-3	3.447	3.739	5.969	BDL	BDL	R	E9D-pH9-3	2.449	2.503	2.495	BDL	BDL
	E9-PH9-B	BDL	BDL	BDL	BDL	BDL							
R	E9-PH9-B	BDL	BDL	BDL	BDL	BDL							
	E9-PH9-C	9.320	8.759	9.018	BDL	BDL							
R	E9-PH9-C	9.185	8.450	8.591	BDL	BDL							
	ACN Wash	BDL	BDL	BDL	BDL	BDL							
	ACN Wash	BDL	BDL	BDL	BDL	BDL							
	ACN Wash	BDL	BDL	BDL	BDL	BDL							
	ACN Wash	BDL	BDL	BDL	BDL	BDL							
	ACN Wash	BDL	BDL	BDL	BDL	BDL							
	ACN Wash	BDL	BDL	BDL	BDL	BDL							
R	ACN Wash	BDL	BDL	BDL	BDL	BDL							
R	ACN Wash	BDL	BDL	BDL	BDL	BDL							
R	ACN Wash	BDL	BDL	BDL	BDL	BDL							
R	ACN Wash	BDL	BDL	BDL	BDL	BDL							
R	ACN Wash	BDL	BDL	BDL	BDL	BDL							
R	ACN Wash	BDL	BDL	BDL	BDL	BDL							
	Calibration	1.000	1.000	1.000	1.000	1.000							
	Calibration	1.000	1.000	1.000	1.000	1.000							
	Calibration	1.000	1.000	1.000	1.000	1.000							
R	Calibration	1.000	1.000	1.000	1.000	1.000							
R	Calibration	1.000	1.000	1.000	1.000	1.000							
R	Calibration	1.000	1.000	1.000	1.000	1.000							

Test 7 - Desorption of Unfired Propellant							Test 7d - Desorption of Unfired Propellant						
		Sorption Aqueous Concentrations C_t (mg/L)							Desorption Aqueous Concentrations C_e (mg/L)				
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN	Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN
							w/o biocide	E11-K7-1UFd	BDL	BDL	0.893	0.165 J	0.112 J
	Not Applicable						R	E11-K7-1UFd	BDL	BDL	0.914	0.161 J	0.114 J
								E11-K7-2UFd	BDL	BDL	1.155	0.207 J	0.148 J
							R	E11-K7-2UFd	BDL	BDL	1.136	0.207 J	0.148 J
								E11-K7-3UFd	BDL	BDL	0.597	0.117 J	0.078 J
							R	E11-K7-3UFd	BDL	BDL	0.596	0.118 J	0.081 J
								E11-K7-Blank	BDL	BDL	BDL	BDL	BDL
							R	E11-K7-Blank	BDL	BDL	BDL	BDL	BDL
								E11-UFP-Blank	BDL	BDL	1.131	BDL	BDL
							R	E11-UFP-Blank	BDL	BDL	1.101	BDL	BDL
							With biocide	T7d-R1	BDL	BDL	10.299	0.24 J	BDL
								T7d-R2	BDL	BDL	10.469	0.301	BDL
								T7d-R3	BDL	BDL	7.917	0.261	BDL
								ACN Wash	BDL	BDL	BDL	BDL	BDL
								ACN Wash	BDL	BDL	BDL	BDL	BDL
								ACN Wash	BDL	BDL	BDL	BDL	BDL
								ACN Wash	BDL	BDL	BDL	BDL	BDL
								ACN Wash	BDL	BDL	BDL	BDL	BDL
								ACN Wash	BDL	BDL	BDL	BDL	BDL
								Cal-Unknown	1.006	0.993	1.001	0.998	1.004
								Calibration	1.000	1.000	1.000	1.000	1.000
								Calibration	1.000	1.000	1.000	1.000	1.000

HPLC Batch Test Data (cont.)

Test 8 - Desorption of Contaminated Soil								Test 8d - Desorption of Contaminated Soil						
		Sorption Aqueous Concentrations C_t (mg/L)								Desorption Aqueous Concentrations C_e (mg/L)				
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN	Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN	
							Kilo range #1 w/o bio.	E10-K1-1d	BDL	BDL	BDL	BDL	BDL	
							R	E10-K1-1d	BDL	BDL	BDL	BDL	BDL	
								E10-K1-2d	BDL	BDL	BDL	BDL	BDL	
							R	E10-K1-2d	BDL	BDL	BDL	BDL	BDL	
								E10-K1-3d	BDL	BDL	BDL	BDL	BDL	
							R	E10-K1-3d	BDL	BDL	BDL	BDL	BDL	
							Kilo range #2 w/o bio.	E10-K2-1d	BDL	BDL	BDL	BDL	BDL	
							R	E10-K2-1d	BDL	BDL	BDL	BDL	BDL	
								E10-k2-2d	BDL	BDL	BDL	BDL	BDL	
							R	E10-k2-2d	BDL	BDL	BDL	BDL	BDL	
								E10-K2-3d	BDL	BDL	BDL	BDL	BDL	
							R	E10-K2-3d	BDL	BDL	BDL	BDL	BDL	
								E10-Blank	BDL	BDL	BDL	BDL	BDL	
							R	E10-Blank	BDL	BDL	BDL	BDL	BDL	
							Kilo range #1 with bio	E10-K1-1d-YB	BDL	BDL	BDL	BDL	BDL	
							R	E10-K1-1d-YB	BDL	BDL	BDL	BDL	BDL	
								E10-K1-2d-YB	BDL	BDL	BDL	BDL	BDL	
							R	E10-K1-2d-YB	BDL	BDL	BDL	BDL	BDL	
								E10-K1-3d-YB	BDL	BDL	BDL	BDL	BDL	
							R	E10-K1-3d-YB	BDL	BDL	BDL	BDL	BDL	
							Kilo range #2 with bio	E10-K2-1d-YB	BDL	BDL	BDL	BDL	BDL	
							R	E10-K2-1d-YB	BDL	BDL	BDL	BDL	BDL	
								E10-K2-2d-YB	BDL	BDL	BDL	BDL	BDL	
							R	E10-K2-2d-YB	BDL	BDL	BDL	BDL	BDL	
								E10-K2-3d-YB	BDL	BDL	BDL	BDL	BDL	
							R	E10-K2-3d-YB	BDL	BDL	BDL	BDL	BDL	
								E10-Blank-YB	BDL	BDL	BDL	BDL	BDL	
							R	E10-Blank-YB	BDL	BDL	BDL	BDL	BDL	
								ACN Wash	BDL	BDL	BDL	BDL	BDL	
								ACN Wash	BDL	BDL	BDL	BDL	BDL	
								ACN Wash	BDL	BDL	BDL	BDL	BDL	
								ACN Wash	BDL	BDL	BDL	BDL	BDL	
								ACN Wash	BDL	BDL	BDL	BDL	BDL	
								ACN Wash	BDL	BDL	BDL	BDL	BDL	
							R	ACN Wash	BDL	BDL	BDL	BDL	BDL	
							R	ACN Wash	BDL	BDL	BDL	BDL	BDL	
							R	ACN Wash	BDL	BDL	BDL	BDL	BDL	
							R	ACN Wash	BDL	BDL	BDL	BDL	BDL	
								ACN Wash	BDL	BDL	BDL	BDL	BDL	
								ACN Wash	BDL	BDL	BDL	BDL	BDL	
							R	ACN Wash	BDL	BDL	BDL	BDL	BDL	
							R	ACN Wash	BDL	BDL	BDL	BDL	BDL	
							R	ACN Wash	BDL	BDL	BDL	BDL	BDL	
							R	ACN Wash	BDL	BDL	BDL	BDL	BDL	
								Calibration	1.000	1.000	1.000	1.000	1.000	
								Calibration	1.100	1.000	1.000	1.000	1.000	
								Calibration	1.000	1.000	1.000	1.000	1.000	
							R	Calibration	1.000	1.000	1.000	1.000	1.000	
							R	Cal-Unknown	1.006	0.993	1.001	0.998	1.004	
								Calibration	1.000	1.000	1.000	1.000	1.000	
								Calibration	1.000	1.000	1.000	1.000	1.000	
							R	Calibration	1.000	1.000	1.000	1.000	1.000	

Not Applicable

HPLC Batch Test Data (cont.)

Test 9 - Repeat of Rainwater Test								Test 9d - Repeat of Rainwater Test						
		Sorption Aqueous Concentrations C_t (mg/L)								Desorption Aqueous Concentrations C_e (mg/L)				
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN	Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN	
w/o biocide	NB-rain-1	3.61	5.1	6.34	0.89	0.81		NB-R1D	0.031 J	0.100 J	BDL	BDL	BDL	
	NB-rain-2	4.34	5.45	7.18	0.89	0.71		NB-R2D	BDL	BDL	BDL	BDL	BDL	
	NB-rain-3	4.3	5.32	6.88	0.91	0.76		NB-R3D	0.121	0.348	BDL	BDL	BDL	
	NB-rain-C	9.89	8.92	9.93	BDL	BDL								
	NB-rain-B	BDL	BDL	0.22 J	0.24 J	0.15 J								
With biocide	YB-rain-1	5.81	5.87	8.45	0.36	BDL		YB-R1D	1.524	1.073	1.136	0.186 J	BDL	
	YB-rain-2	5.6	5.59	8.52	BDL	BDL		YB-R2D	1.410	0.939	0.995	0.179 J	BDL	
	YB-rain-3	5.62	5.58	8.32	BDL	BDL		YB-R3D	1.475	1.024	1.027	0.202 J	BDL	
	YB-rain-C	10.01	9.25	9.83	BDL	BDL								
	YB-rain-B	BDL	0.19 J	0.29	BDL	BDL								
	ACN Wash	BDL	BDL	0.29	BDL	BDL								
	ACN Wash	BDL	0.24	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	0.27	BDL	BDL	BDL								
	Calibration	10	10	10	10	10								
	Calibration	10	10	10	10	10								
	NG Std (MEW)	BDL	0.19 J	10.51	BDL	BDL								

Test 10 - Fired Propellant								Test 10d - Fired Propellant						
		Sorption Aqueous Concentrations C_t (mg/L)								Desorption Aqueous Concentrations C_e (mg/L)				
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN		Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN
								With biocide	T12A-R1	BDL	BDL	28.861	1.049	0.572
									T12A-R2	BDL	BDL	28.648	1.145	0.629
		Not Applicable							T12A-R3	BDL	BDL	29.817	1.157	0.616
								w/o biocide	T12B-R1	BDL	BDL	19.482	3.672	2.711
									T12B-R2	BDL	BDL	17.448	4.242	3.024
									T12B-R3	BDL	BDL	16.277	3.925	2.585
									Calibration	1.000	1.000	1.000	1.000	1.000
									Calibration	1.000	1.000	1.000	1.000	1.000
									Calibration	1.000	1.000	1.000	1.000	1.000
									Cal-Unknown	1.006	0.995	1.007	0.991	0.998
									NG Std (MEW)	BDL	BDL	0.996	BDL	BDL
									NG Std (MEW)	BDL	BDL	1.025	BDL	BDL
									ACN Wash	BDL	BDL	BDL	BDL	BDL
									ACN Wash	BDL	BDL	BDL	BDL	BDL
									ACN Wash	BDL	BDL	BDL	BDL	BDL
									ACN Wash	BDL	BDL	BDL	BDL	BDL
									ACN Wash	BDL	BDL	BDL	BDL	BDL
									ACN Wash	BDL	BDL	BDL	BDL	BDL
									ACN Wash	BDL	BDL	BDL	BDL	BDL

HPLC Batch Test Data (cont.)

Pre-Test 4 - Biocide Comparison								Pre-Test 4d - Biocide Comparison						
		Sorption Aqueous Concentrations C_t (mg/L)								Desorption Aqueous Concentrations C_e (mg/L)				
Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN		Other	Sample	2,4-DNT	2,6-DNT	NG	1,2-GDN	1,3-GDN
Glutaraldehyde	T13-Control	9.800	10.670	10.390	BDL	BDL			T13D-R1	1.270	0.926	0.484	0.259	BDL
	T13-R1	7.870	7.370	8.970	0.220	J BDL			T13D-R2	1.130	0.833	0.431	0.243	J BDL
	T13-R2	8.350	7.720	8.960	BDL	BDL			T13D-R3	1.122	0.841	0.434	0.203	J BDL
	T13-R3	8.120	7.570	8.970	0.210	J BDL								
HgCl	T14-Control	10.070	8.970	9.530	BDL	BDL			T14D-R1	0.812	0.916	0.350	0.202	J 0.085 J
	T14-R1	7.560	7.110	8.960	BDL	BDL			T14D-R2	0.874	0.915	0.286	0.130	J 0.066 J
	T14-R2	7.960	7.290	9.050	BDL	BDL			T14D-R3	0.874	0.993	0.375	0.174	J 0.080 J
	T14-R3	7.710	6.970	8.910	BDL	BDL								
	Calibration	1.000	1.000	1.000	1.000	1.000								
	Calibration	1.000	1.000	1.000	1.000	1.000								
	Calibration	1.000	1.000	1.000	1.000	1.000								
	Cal-Unknown	1.006	0.995	1.007	0.991	0.998								
	NG Std (MEW)	BDL	BDL	0.996	BDL	BDL								
	NG Std (MEW)	BDL	BDL	1.025	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								
	ACN Wash	BDL	BDL	BDL	BDL	BDL								

Abbreviations
Sample ID-C = Control = Initial spike concentration (C_o)
Sample ID-B = Blank = DI water
R = Reverse order (some sets of samples were analyzed in both forward and reverse order on the HPLC)
dup = analytical duplicate
NG = nitroglycerin
2,4-DNT = 2,4-dinitrotoluene
2,6-DNT = 2,6-dinitrotoluene
1,2-GDN = glycerol-1,2-dinitrate
1,3-GDN = glycerol-1,3-dinitrate
BDL = below detection limit
J = estimated value

Appendix G

Batch test partitioning coefficient (K_d) calculations.

Test 1 - Equilibration Time						Test 1d - Equilibration Time						Corrected		
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)		
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG
0 hours	E4-000-1	4.0	3.0	1.0		0 hours	E4D-000-1	11.7	7.2	0.1		13.0	8.3	1.6
	E4-000-2	5.6	4.1	1.3			E4D-000-2	21.0	14.8	3.5		22.3	16.2	5.4
	E4-000-3	3.4	2.5	0.8			E4D-000-3	11.9	6.8	0.0		13.3	8.2	1.9
						R	E4D-000-1	12.4	7.2	0.1		13.7	8.4	1.5
						R	E4D-000-2	21.8	14.7	3.5		23.2	16.1	5.4
						R	E4D-000-3	12.4	6.9	0.0		13.9	8.3	1.9
24 hours	E4-024-1	4.0	3.0	0.9		24 hours	E4D-024-1	4.6	2.0	-2.3		5.3	2.7	-1.5
	E4-024-2	4.0	2.9	1.0			E4D-024-2	4.5	2.0	-2.1		5.2	2.8	-1.3
	E4-024-3	4.3	3.2	1.2		dup	E4D-024-2	4.5	2.0	-2.1		5.2	2.7	-1.3
							E4D-024-3	5.4	2.9	-1.2		6.2	3.7	-0.2
						R	E4D-024-1	4.8	2.0	-2.3		5.6	2.7	-1.5
						R	E4D-024-2	4.7	2.1	-2.1		5.4	2.8	-1.3
						R dup	E4D-024-2	4.7	2.1	-2.1		5.4	2.8	-1.3
						R	E4D-024-3	5.4	2.9	-1.2		6.1	3.6	-0.3
48 hours	E4-048-1	4.2	3.1	1.1		48 hours	E4D-048-1	5.3	3.0	-0.9		6.1	3.8	0.4
dup	E4-048-1	3.9	2.9	0.9			E4D-048-2	4.7	2.1	-0.5		5.4	2.8	0.6
	E4-048-2	4.0	2.7	1.1			E4D-048-3	4.6	1.9	-1.8		5.4	2.7	-0.7
	E4-048-3	3.7	2.6	0.8		R	E4D-048-1	5.5	3.1	-0.9		6.3	3.9	0.4
						R	E4D-048-2	4.9	2.1	-0.6		5.6	2.9	0.6
						R	E4D-048-3	4.8	1.9	-1.8		5.6	2.7	-0.7
72 hours	E4-072-1	4.2	3.2	1.1		72 hours	E4D-072-1	5.5	4.2	0.2		6.2	5.0	1.6
	E4-072-2	4.2	3.2	1.1			E4D-072-2	5.2	4.1	-0.3		5.9	5.0	1.0
	E4-072-3	4.0	3.0	1.0			E4D-072-3	4.6	3.6	-0.1		5.3	4.4	1.3
						R	E4D-072-1	5.6	4.3	0.2		6.3	5.1	1.6
						R	E4D-072-2	5.2	4.3	-0.2		5.9	5.1	1.1
						R	E4D-072-3	4.6	3.7	0.0		5.4	4.6	1.5
120 hours	E4-120-1	4.1	3.1	1.1		120 hours	E4D-120-1	4.5	4.1	-1.1		5.2	4.9	-0.1
	E4-120-2	4.2	3.0	1.0			E4D-120-2	4.7	3.8	-1.2		5.4	4.7	-0.1
	E4-120-3	4.1	2.9	0.9			E4D-120-3	4.3	3.5	-1.9		5.0	4.4	-0.9
						R	E4D-120-1	4.5	3.9	-1.1		5.2	4.7	0.0
						R	E4D-120-2	4.8	3.8	-1.1		5.5	4.6	0.0
						R	E4D-120-3	4.2	3.4	-1.9		4.9	4.2	-0.8
216 hours	E4-216-1	4.5	3.1	1.2										
dup	E4-216-1	4.6	3.4	1.3		240 hours	E4D-240-1	5.4	3.7	0.3		6.1	4.5	1.5
	E4-216-2	4.5	3.3	1.3			E4D-240-2	5.3	4.0	0.2		5.9	4.8	1.4
	E4-216-3	4.5	3.2	1.3			E4D-240-3	5.5	3.8	0.6		6.1	4.6	1.9

Batch test partitioning coefficient (K_d) calculations (cont.).

Test 2 - NG/DNT Concentration						Test 2d - NG/DNT Concentration						Corrected					
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)					
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG			
0.1 ppm	E5-010-1	5.70	3.05	0.67			E5D-010-1	-1.2	-3.0	-4.7		-1.0	-2.9	-4.4			
dup	E5-010-1	5.95	4.13	0.30			E5D-010-2	5.0	0.6	BDL		5.5	1.1	BDL			
	E5-010-2	5.45	3.19	0.09			E5D-010-3	1.3	1.4	BDL		1.6	1.8	BDL			
	E5-010-3	5.95	3.96	-0.15													
1.0 ppm	E5-100-1	4.95	3.64	0.95			E5D-100-1	5.7	3.3	-1.6		6.4	4.0	-0.5			
	E5-100-2	4.55	3.40	0.87			E5D-100-2	5.9	3.5	-1.5		6.6	4.2	-0.3			
	E5-100-3	4.69	3.48	0.90			E5D-100-3	5.7	3.2	-1.3		6.3	3.9	-0.1			
40.0 ppm	E5-400-1	3.26	2.72	0.96			E5D-400-1	3.9	4.0	-1.2		4.7	5.0	-0.1			
	E5-400-2	3.22	2.64	0.97			E5D-400-2	4.1	3.5	-0.2		4.9	4.5	1.2			
	E5-400-3	3.41	2.75	0.93			E5D-400-3	4.3	3.6	-0.5		5.1	4.6	0.9			
80.0 ppm	E5-800-1	2.80	2.36	0.79			E5D-800-1	2.1	2.6	-2.5		2.8	3.5	-1.6			
	E5-800-2	2.84	2.36	0.84			E5D-800-2	2.9	2.5	-1.1		3.8	3.4	0.3			
	E5-800-3	2.82	2.35	0.88			E5D-800-3	3.0	2.4	-1.0		3.8	3.3	0.4			
R	E5-010-1	4.02	5.80	0.67													
R dup	E5-010-1	5.00	2.92	0.75		R	E5D-010-1	-1.9	-3.3	-4.2		-1.7	-3.2	-3.9			
R	E5-010-2	2.67	2.54	0.59		R	E5D-010-2	-0.2	-1.1	BDL		0.4	-0.6	BDL			
R	E5-010-3	4.20	3.05	1.10		R	E5D-010-3	3.8	-0.7	BDL		4.4	-0.3	BDL			
R	E5-100-1	4.97	3.94	0.78		R	E5D-100-1	5.1	3.4	-2.1		5.7	4.0	-1.0			
R	E5-100-2	4.45	3.29	0.81		R	E5D-100-2	5.5	3.2	-1.9		6.2	3.9	-0.7			
R	E5-100-3	4.82	3.29	0.52		R	E5D-100-3	6.9	2.8	-2.7		7.6	3.5	-1.4			
R	E5-400-1	3.23	2.80	0.97		R	E5D-400-1	3.8	4.2	-1.2		4.6	5.2	-0.1			
R	E5-400-2	3.16	2.75	0.97		R	E5D-400-2	3.9	3.7	-0.4		4.8	4.6	1.0			
R	E5-400-3	3.40	2.85	0.93		R	E5D-400-3	4.3	3.8	-0.6		5.1	4.7	0.8			
R	E5-800-1	2.81	2.37	0.85		R	E5D-800-1	2.1	2.6	-2.4		2.9	3.5	-1.5			
R	E5-800-2	2.88	2.32	0.85		R	E5D-800-2	3.0	2.4	-1.0		3.8	3.3	0.3			
R	E5-800-3	2.89	2.34	0.91		R	E5D-800-3	3.1	2.4	-0.9		4.0	3.4	0.4			

Test 3 - Surface Soil Heterogeneity						Test 3d - Surface Soil Heterogeneity						Corrected					
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)					
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG			
Echo range #1	E6-E1-1	2.1	1.9	0.7			E6D-E1-1	4.6	4.2	1.1		5.9	5.7	3.7			
R	E6-E1-1	2.0	1.7	0.6		R	E6D-E1-1	4.1	3.5	0.6		5.4	5.0	3.2			
	E6-E1-2	1.9	1.6	0.7			E6D-E1-2	3.6	2.4	0.8		5.2	3.9	3.8			
R	E6-E1-2	1.8	1.4	0.7		R	E6D-E1-2	3.5	1.5	1.0		5.0	3.1	3.9			
	E6-E1-3	1.9	1.6	0.6			Rep 3 Soils Sacrificed for Soil Analysis										
dup	**E6-E1-3**	1.9	1.6	0.7			Rep 3 Soils Sacrificed for Soil Analysis										
R	**E6-E1-3**	1.9	1.5	0.7			Rep 3 Soils Sacrificed for Soil Analysis										
R dup	**E6-E1-3**	1.8	1.4	0.8			Rep 3 Soils Sacrificed for Soil Analysis										
Echo range #2	E6-E2-1	1.8	1.5	0.6			E6D-E2-1	3.8	3.4	-0.2		5.3	5.0	2.3			
R	E6-E2-1	1.8	1.4	0.5		R	E6D-E2-1	3.8	2.8	-0.4		5.3	4.5	2.2			
	E6-E2-2	1.7	1.5	0.6			E6D-E2-2	3.6	2.2	1.1		4.0	2.5	1.8			
R	E6-E2-2	1.7	1.4	0.6		R	E6D-E2-2	3.4	1.8	0.6		3.7	2.1	1.3			
	E6-E2-3	1.9	1.5	0.6			Rep 3 Soils Sacrificed for Soil Analysis										
R	E6-E2-3	2.0	1.5	0.8			Rep 3 Soils Sacrificed for Soil Analysis										
Juliet range #1	E6-J1-1	2.1	1.7	0.6			E6D-J1-1	4.3	3.0	-0.4		5.6	4.4	1.8			
R	E6-J1-1	2.0	1.8	0.5		R	E6D-J1-1	4.0	3.2	-1.1		5.3	4.6	1.1			
	E6-J1-2	2.2	1.8	0.6			E6D-J1-2	4.0	2.3	0.0		5.0	3.3	1.9			
dup	**E6-J1-2**	2.3	1.8	0.9		R	E6D-J1-2	4.2	2.3	1.6		5.1	3.4	3.4			
R	**E6-J1-2**	2.2	1.8	0.7													
R dup	**E6-J1-2**	2.2	1.7	0.7													
	E6-J1-3	2.3	1.9	0.7			Rep 3 Soils Sacrificed for Soil Analysis										
R	E6-J1-3	2.4	1.8	0.6			Rep 3 Soils Sacrificed for Soil Analysis										
Juliet range #2	E6-J2-1	3.2	2.6	1.0			E6D-J2-1	7.5	5.0	0.9		8.6	6.1	2.5			
R	E6-J2-1	3.4	2.6	1.0		R	E6D-J2-1	7.7	5.0	0.7		8.8	6.1	2.3			
	E6-J2-2	3.4	2.8	0.9			E6D-J2-2	5.5	4.5	0.3		7.4	6.6	3.9			
R	E6-J2-2	3.4	2.7	1.0		R	E6D-J2-2	5.4	4.2	0.4		7.4	6.4	4.0			
	E6-J2-3	3.6	2.9	1.0			Rep 3 Soils Sacrificed for Soil Analysis										
R	E6-J2-3	3.7	2.7	1.1			Rep 3 Soils Sacrificed for Soil Analysis										
Kilo range,west	E6-K6-1	2.6	2.2	0.9			E6D-K6-1	4.7	3.6	0.0		5.8	4.7	1.7			
R	E6-K6-1	2.7	2.1	0.8		R	E6D-K6-1	4.9	3.2	-0.4		5.9	4.4	1.3			
	E6-K6-2	2.8	2.4	1.0			E6D-K6-2	4.0	2.9	0.4		5.4	4.3	2.7			
R	E6-K6-2	2.9	2.1	0.8		R	E6D-K6-2	4.0	2.2	-0.3		5.4	3.6	2.1			
	E6-K6-3	2.9	2.5	0.9			Rep 3 Soils Sacrificed for Soil Analysis										
R	E6-K6-3	3.0	2.3	0.8			Rep 3 Soils Sacrificed for Soil Analysis										

Batch test partitioning coefficient (K_d) calculations (cont.).

Test 4 - Depth Evaluation						Test 4d - Depth Evaluation						Corrected		
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)		
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG
Kilo range 9-12"	E7-K3-1	1.3	0.9	0.5			E7D-K3-1	3.5	-1.4	-3.0		5.7	0.0	-1.5
R	E7-K3-1	1.3	1.0	0.4		R	E7D-K3-1	3.3	-0.9	-3.0		5.4	0.4	-1.5
	E7-K3-2	1.3	0.8	0.3			E7D-K3-2	3.3	-1.6	-3.5		5.1	-0.4	-2.0
R	E7-K3-2	1.3	0.8	0.3		R	E7D-K3-2	3.4	-1.5	-3.2		5.3	-0.3	-1.8
	E7-K3-3	1.3	0.9	0.3			E7D-K3-3	3.0	-1.4	-3.5		5.1	0.0	-2.0
R	E7-K3-3	1.2	0.8	0.4		R	E7D-K3-3	2.4	-1.5	-3.3		4.5	-0.2	-1.8
Kilo range 18-24"	E7-K4-1	0.9	0.6	0.3			E7D-K4-1	3.7	-2.4	-4.0		4.1	-2.2	-3.7
dup	E7-K4-1	0.9	0.5	0.3		R	E7D-K4-1	3.5	-2.6	-3.9		4.0	-2.4	-3.7
R	E7-K4-1	0.9	0.5	0.2			E7D-K4-2	2.5	-3.0	-4.2		5.6	-1.3	-2.7
R dup	E7-K4-1	0.8	0.5	0.2		R	E7D-K4-2	2.4	-3.0	-4.4		5.5	-1.3	-2.9
	E7-K4-2	0.8	0.4	0.2			E7D-K4-3	3.6	-2.8	-4.2		6.5	-1.3	-2.8
R	E7-K4-2	0.8	0.4	0.1		R	E7D-K4-3	3.5	-2.9	-3.8		6.5	-1.4	-2.5
	E7-K4-3	1.0	0.5	0.2										
R	E7-K4-3	1.0	0.5	0.3										
Kilo range 30-36"	E7-K5-1	0.7	0.4	0.3			E7D-K5-1	4.1	-2.9	-3.9		7.4	-1.6	-2.9
R	E7-K5-1	0.7	0.4	0.2		R	E7D-K5-1	4.3	-2.7	-4.0		7.5	-1.5	-2.9
	E7-K5-2	0.7	0.3	0.3			E7D-K5-2	4.9	-3.3	-3.9		7.6	-2.2	-3.0
R	E7-K5-2	0.7	0.4	0.2		R	E7D-K5-2	3.8	-3.0	-4.0		6.5	-1.9	-3.2
	E7-K5-3	0.6	0.3	0.2			E7D-K5-3	4.0	-3.4	-4.1		5.9	-2.7	-3.5
R	E7-K5-3	0.6	0.2	0.2		R	E7D-K5-3	3.7	-3.7	-4.2		5.5	-3.0	-3.7
Echo range 9-12"	E7-E3-1	1.3	1.0	0.2			E7D-E3-1	4.8	8.6	1.4		6.8	11.1	6.7
R	E7-E3-1	1.3	1.0	0.2		R	E7D-E3-1	5.0	9.1	0.8		7.0	11.6	6.2
	E7-E3-2	1.2	0.8	0.1			E7D-E3-2	4.7	8.3	0.1		6.6	10.7	5.2
R	E7-E3-2	1.2	0.8	0.4		R	E7D-E3-2	4.6	7.8	4.2		6.6	10.3	8.7
	E7-E3-3	1.3	0.9	0.2			E7D-E3-3	4.8	8.4	1.4		6.8	10.9	6.7
R	E7-E3-3	1.2	0.8	0.2		R	E7D-E3-3	4.6	8.2	1.5		6.7	10.8	6.7
Echo range 18-24"	E7-E4-1	0.6	0.6	0.3			E7D-E4-1	3.0	10.5	5.1		5.4	13.4	10.1
R	E7-E4-1	0.6	0.5	0.3		R	E7D-E4-1	3.0	9.8	4.1		5.4	12.8	9.0
	E7-E4-2	0.5	0.5	-0.2			E7D-E4-2	2.4	9.7	-5.9		4.7	12.4	-0.1
R	E7-E4-2	0.6	0.5	0.1		R	E7D-E4-2	3.0	9.7	0.5		5.3	12.5	5.8
	E7-E4-3	0.5	0.5	0.1			E7D-E4-3	2.2	9.5	0.4		4.3	11.9	5.6
R	E7-E4-3	0.6	0.4	0.2		R	E7D-E4-3	2.5	8.1	4.2		4.6	10.6	9.1
Echo range 30-36	E7-E5-1	0.7	0.5	0.0			E7D-E5-1	3.4	8.8	-1.9		5.7	11.7	3.1
R	E7-E5-1	0.8	0.5	0.2		R	E7D-E5-1	4.5	8.7	3.1		6.8	11.6	8.1
	E7-E5-2	0.9	0.7	0.2			E7D-E5-2	5.1	11.2	2.1		7.4	14.0	7.1
R	E7-E5-2	0.9	0.6	0.2		R	E7D-E5-2	5.5	10.1	3.5		8.3	12.9	8.5
	E7-E5-3	0.9	0.7	0.0			E7D-E5-3	4.5	9.4	-1.3		6.8	12.2	3.8
R	E7-E5-3	1.1	0.8	0.1		R	E7D-E5-3	5.6	10.7	0.4		7.8	13.5	5.6
Juliet range 9-12"	E7-J3-1	0.7	0.6	0.0			E7D-J3-1	1.7	8.2	-2.4		4.7	12.3	3.5
R	E7-J3-1	0.8	0.4	0.3		R	E7D-J3-1	2.2	5.8	1.7		5.2	10.2	7.6
	E7-J3-2	0.8	0.7	0.1			E7D-J3-2	2.6	8.9	-0.9		5.1	12.3	4.9
R	E7-J3-2	0.7	0.5	0.2		R	E7D-J3-2	2.0	7.5	1.0		4.4	10.9	6.6
	E7-J3-3	0.9	0.7	0.1			E7D-J3-3	2.5	8.7	-0.7		5.2	12.5	5.0
R	E7-J3-3	1.0	0.7	0.2		R	E7D-J3-3	2.9	8.3	0.5		5.6	12.1	6.5
Juliet range 18-24"	E7-J4-1	0.7	0.2	0.0			E7D-J4-1	2.2	9.7	-1.7		5.3	16.3	4.6
R	E7-J4-1	0.6	0.2	-0.1		R	E7D-J4-1	1.7	10.9	-4.4		4.8	17.5	2.1
	E7-J4-2	0.6	0.3	0.0			E7D-J4-2	1.6	11.7	-2.5		4.9	19.0	4.0
R	E7-J4-2	0.6	0.2	0.0		R	E7D-J4-2	1.9	10.9	-2.5		5.2	18.0	4.1
	E7-J4-3	0.6	0.2	-0.1			E7D-J4-3	1.8	9.7	-4.3		4.9	16.1	2.4
R	E7-J4-3	0.6	0.2	0.0		R	E7D-J4-3	1.8	10.2	-1.7		5.0	16.7	4.8
Juliet range 30-36"	E7-J5-1	0.2	0.2	0.1			E7D-J5-1	0.9	20.6	0.3		5.3	28.0	6.3
R	E7-J5-1	0.2	0.1	0.1		R	E7D-J5-1	0.8	19.0	0.0		5.3	27.3	5.8
	E7-J5-2 repeat	0.3	0.2	0.0			E7D-J5-2 repeat	-0.4	16.8	-4.5		4.1	25.2	0.2
R	E7-J5-2 repeat	0.1	0.2	-0.1		R	E7D-J5-2 repeat	0.8	18.1	-4.1		5.3	26.8	0.5
R dup	E7-J5-2 repeat	0.2	0.2	-0.1			E7D-J5-3 repeat	0.7	4.5	-2.9		6.0	15.3	3.6
	E7-J5-3 repeat	0.2	-0.3	-0.1		R	E7D-J5-3 repeat	2.6	23.4	0.0		7.8	33.1	6.3
R	E7-J5-3 repeat	0.3	0.2	0.1										

Batch test partitioning coefficient (K_d) calculations (cont.).

Test 5 - Temperature Evaluation						Test 5d - Temperature Evaluation						Corrected		
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)		
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG
12ºC	E8-T12-1	4.1	3.1	0.9			E8D-T12-1	6.6	5.8	-1.8		7.4	6.9	-0.8
dup	E8-T12-1	4.2	3.2	0.9			E8D-T12-2	6.8	6.2	-1.5		7.5	7.1	-0.5
	E8-T12-2	5.1	3.8	1.0			E8D-T12-3	6.6	5.9	-1.7		7.3	6.8	-0.8
	E8-T12-3	4.9	3.6	1.0										
32ºC	E8-T32-1	3.3	2.5	0.9			E8D-T32-1	2.8	2.3	-2.2		3.5	3.2	-1.3
	E8-T32-2	3.2	2.3	0.9			E8D-T32-2	4.0	3.4	-1.8		4.9	4.4	-0.8
	E8-T32-3	3.5	2.5	1.0			E8D-T32-3	3.5	2.5	-1.7		4.3	3.4	-0.7
R	E8-T12-1	4.0	3.0	0.9		R	E8D-T12-1	6.3	5.2	-1.9		7.2	6.2	-0.9
R dup	E8-T12-1	4.1	3.0	0.9		R	E8D-T12-2	6.8	5.7	-1.7		7.5	6.5	-0.7
R	E8-T12-2	5.0	3.6	1.0		R	E8D-T12-3	6.6	5.7	-2.1		7.3	6.5	-1.2
R	E8-T12-3	4.9	3.6	0.9										
R	E8-T32-1	3.3	2.3	0.9		R	E8D-T32-1	2.8	1.9	-2.3		3.5	2.8	-1.4
R	E8-T32-2	3.3	2.3	1.0		R	E8D-T32-2	4.1	3.3	-1.5		4.9	4.4	-0.5
R	E8-T32-3	3.5	2.4	0.9		R	E8D-T32-3	3.5	2.3	-2.2		4.2	3.2	-1.2

Test 6 - pH						Test 6d - pH						Corrected		
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)		
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG
pH=4.2	E9-pH4-1	6.0	6.3	2.9			E9D-PH4-1	5.5	10.0	0.5		5.5	10.0	0.5
Dup	E9-pH4-1	6.1	6.3	3.1		R	E9D-PH4-1	5.6	10.2	0.9		5.6	10.2	0.9
R	E9-pH4-1	6.1	6.2	3.1			E9D-PH4-2	5.3	9.6	0.5		5.3	9.6	0.5
R dup	E9-pH4-1	6.2	6.5	3.3		R	E9D-PH4-2	5.4	9.7	0.7		5.4	9.7	0.7
	E9-pH4-2	6.1	6.3	3.1			E9D-PH4-3	5.7	9.7	1.0		5.7	9.7	1.0
R	E9-pH4-2	6.2	6.3	3.2		R	E9D-PH4-3	5.9	10.2	1.1		5.9	10.2	1.1
	E9-pH4-3	6.1	6.3	3.1										
R	E9-pH4-3	6.3	6.7	3.0										
pH=8.2	E9-PH9-1	8.7	5.9	2.2			E9D-pH9-1	5.8	5.6	0.9		5.8	5.6	0.9
R	E9-PH9-1	9.0	6.7	2.4		R	E9D-pH9-1	6.0	6.0	1.2		6.0	6.0	1.2
	E9-PH9-2	7.8	5.6	2.0			E9D-pH9-2	5.4	5.5	0.8		5.4	5.5	0.8
R	E9-PH9-2	8.1	5.7	2.2		R	E9D-pH9-2	5.7	5.7	1.1		5.7	5.7	1.1
	E9-PH9-3	8.1	6.5	2.1			E9D-pH9-3	5.3	5.9	0.8		5.3	5.9	0.8
R	E9-PH9-3	8.4	6.5	2.4		R	E9D-pH9-3	5.4	5.8	1.3		5.4	5.8	1.3

Test 7 - Desorption of Unfired Propellant						Test 7d - Desorption of Unfired Propellant						Corrected		
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)		
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG
						w/o biocide	E11-K7-1UFd	BDL	BDL	1249.2				
Not Applicable						R	E11-K7-1UFd	BDL	BDL	1220.4				
							E11-K7-2UFd	BDL	BDL	964.7				
						R	E11-K7-2UFd	BDL	BDL	980.9				
							E11-K7-3UFd	BDL	BDL	1871.0				
						R	E11-K7-3UFd	BDL	BDL	1874.2				
						With biocide	T7d-R1	BDL	BDL	103.7				
							T7d-R2	BDL	BDL	102.0				
							T7d-R3	BDL	BDL	136.5				

Batch test partitioning coefficient (K_d) calculations (cont.).

Test 8 - Desorption of Contaminated Soil						Test 8d - Desorption of Contaminated Soil						Corrected		
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)		
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG
						Kilo range #1 w/o bio.	E10-K1-1d			BDL				
	Not Applicable					R	E10-K1-1d			BDL				
							E10-K1-2d			BDL				
						R	E10-K1-2d			BDL				
							E10-K1-3d			BDL				
						R	E10-K1-3d			BDL				
						Kilo range #2 w/o bio.	E10-K2-1d			BDL				
						R	E10-K2-1d			BDL				
							E10-k2-2d			BDL				
						R	E10-k2-2d			BDL				
							E10-K2-3d			BDL				
						R	E10-K2-3d			BDL				
						Kilo range #1 with bio.	E10-K1-1d-YB			BDL				
						R	E10-K1-1d-YB			BDL				
							E10-K1-2d-YB			BDL				
						R	E10-K1-2d-YB			BDL				
							E10-K1-3d-YB			BDL				
						R	E10-K1-3d-YB			BDL				
						Kilo range #2 with bio.	E10-K2-1d-YB			BDL				
						R	E10-K2-1d-YB			BDL				
							E10-K2-2d-YB			BDL				
						R	E10-K2-2d-YB			BDL				
							E10-K2-3d-YB			BDL				
						R	E10-K2-3d-YB			BDL				

Test 9 - Repeat of Rainwater Test						Test 9d - Repeat of Rainwater Test						Corrected		
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)		
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG
w/o biocide	NB-rain-1	8.8	3.9	2.8			NB-R1D	1018	194	BDL		1052	209	BDL
	NB-rain-2	6.5	3.3	1.9			NB-R2D	BDL	BDL	535		BDL	BDL	619
	NB-rain-3	6.6	3.5	2.2			NB-R3D	228	49	BDL		239	54	BDL
With biocide	YB-rain-1	3.6	2.7	0.8			YB-R1D	8.6	10.0	1.3		9.7	11.6	3.5
	YB-rain-2	3.9	3.1	0.8			YB-R2D	10.4	13.6	1.8		11.6	15.4	4.3
	YB-rain-3	3.9	3.1	0.9			YB-R3D	9.7	12.1	2.6		10.8	13.7	5.0

Test 10 - Fired Propellant						Test 10d - Fired Propellant						Corrected		
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)		
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG
						With biocide	T12A-R1	BDL	BDL	33.8				
	Not Applicable						T12A-R2	BDL	BDL	34.1				
							T12A-R3	BDL	BDL	32.6				
						w/o biocide	T12B-R1	BDL	BDL	52.5				
							T12B-R2	BDL	BDL	59.2				
							T12B-R3	BDL	BDL	63.8				

Pre-Test 4 - Biocide Comparison						Pre-Test 4d - Biocide Comparison						Corrected		
		Sorption Calculation (L/kg)						Desorption Calculation (L/kg)				Desorption Calculation (L/kg)		
		Kd	Kd	Kd				Kd	Kd	Kd		Kd	Kd	Kd
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG		2,4-DNT	2,6-DNT	NG
Glutaraldehyde	T13-R1	1.3	1.7	0.6			T13D-R1	3.1	8.2	5.2		4.9	10.6	10.7
	T13-R2	0.9	1.4	0.6			T13D-R2	2.0	7.6	6.6		4.2	10.3	12.7
	T13-R3	1.1	1.5	0.6			T13D-R3	3.1	8.4	6.4		5.2	11.0	12.5
HgCl	T14-R1	1.6	1.9	0.6			T14D-R1	9.6	9.8	9.3		12.4	12.1	16.8
	T14-R2	1.2	1.7	0.5			T14D-R2	6.3	8.8	10.9		9.0	11.2	20.2
	T14-R3	1.4	2.0	0.6			T14D-R3	7.7	9.4	9.0		10.3	11.4	16.0

Abbreviations

Sample ID-C = Control = Initial spike concentration (C_o)
Sample ID-B = Blank = DI water
R = Reverse order (some sets of samples were analyzed in both forward and reverse order on the HPLC)
dup = analytical duplicate
NG = nitroglycerin
2,4-DNT = 2,4-dinitrotoluene
2,6-DNT = 2,6-dinitrotoluene
BDL = below detection limit

Appendix H

Estimated soil concentration results at the end of the batch adsorption and desorption tests.

Test 1 - Equilibration Time						Test 1 - Equilibration Time				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
0 hours	E4-000-1	20.16	16.84	7.76		0 hours	E4D-000-1	15.6	11.6	2.4
	E4-000-2	24.01	20.34	9.61			E4D-000-2	20.6	16.6	6.1
	E4-000-3	18.41	14.89	6.16			E4D-000-3	14.5	10.3	2.3
						R	E4D-000-1	15.6	12.1	1.8
						R	E4D-000-2	20.6	17.2	5.4
						R	E4D-000-3	14.5	10.9	1.7
24 hours	E4-024-1	20.21	16.89	7.36		24 hours	E4D-024-1	11.1	6.5	-4.0
	E4-024-2	20.16	16.69	7.71			E4D-024-2	11.0	6.5	-3.4
	E4-024-3	21.06	17.49	8.66		dup	E4D-024-2	11.0	6.5	-3.4
							E4D-024-3	12.4	8.1	-0.6
						R	E4D-024-1	11.4	7.1	-4.6
						R	E4D-024-2	11.3	7.1	-4.0
						R dup	E4D-024-2	11.3	7.1	-4.1
						R	E4D-024-3	12.3	8.6	-1.3
48 hours	E4-048-1	20.71	17.34	8.21		48 hours	E4D-048-1	11.7	7.9	0.6
dup	E4-048-1	19.91	16.44	7.11			E4D-048-2	11.2	6.4	1.2
	E4-048-2	20.11	15.89	8.46			E4D-048-3	10.9	6.0	-1.6
	E4-048-3	19.41	15.29	6.71		R	E4D-048-1	12.0	7.9	0.7
						R	E4D-048-2	11.4	6.4	1.1
						R	E4D-048-3	11.1	6.0	-1.5
72 hours	E4-072-1	20.81	17.49	8.31		72 hours	E4D-072-1	12.3	9.6	2.5
	E4-072-2	20.81	17.64	8.31			E4D-072-2	12.0	9.6	1.7
	E4-072-3	20.11	16.89	7.81			E4D-072-3	11.1	8.7	2.1
						R	E4D-072-1	12.4	9.7	2.6
						R	E4D-072-2	12.1	9.7	1.8
						R	E4D-072-3	11.2	8.8	2.3
120 hours	E4-120-1	20.51	17.09	8.41						
	E4-120-2	20.76	16.99	7.91		120 hours	E4D-120-1	11.2	9.3	-0.2
	E4-120-3	20.41	16.64	6.76			E4D-120-2	11.6	9.0	-0.2
							E4D-120-3	10.9	8.5	-1.9
						R	E4D-120-1	11.2	9.1	-0.1
						R	E4D-120-2	11.6	8.9	0.0
						R	E4D-120-3	10.8	8.4	-1.8
216 hours	E4-216-1	21.66	17.39	9.16						
	E4-216-1	21.91	18.24	9.81		240 hours	E4D-240-1	12.6	9.0	2.6
	E4-216-2	21.51	17.84	9.61			E4D-240-2	12.6	9.7	2.5
dup	E4-216-3	21.51	17.69	9.26			E4D-240-3	12.6	9.3	3.1

Test 2 - NG/DNT Concentration						Test 2 - NG/DNT Concentration				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
0.1 ppm	E5-010-1	0.25	0.18	0.05			E5D-010-1	-0.1	-0.3	-0.3
dup	E5-010-1	0.25	0.22	0.02			E5D-010-2	0.1	0.0	BDL
	E5-010-2	0.24	0.19	0.01			E5D-010-3	0.1	0.1	BDL
	E5-010-3	0.25	0.21	-0.01						
1.0 ppm	E5-100-1	2.22	1.92	0.72			E5D-100-1	1.3	0.9	-0.1
	E5-100-2	2.13	1.85	0.67			E5D-100-2	1.3	0.9	-0.1
	E5-100-3	2.16	1.87	0.69			E5D-100-3	1.3	0.9	0.0
40.0 ppm	E5-400-1	72.88	65.79	30.19			E5D-400-1	38.4	36.3	-0.4
	E5-400-2	72.43	64.49	30.54			E5D-400-2	39.3	33.8	7.6
	E5-400-3	74.98	66.34	29.29			E5D-400-3	41.3	35.0	5.8
80.0 ppm	E5-800-1	130.73	117.23	50.58			E5D-800-1	52.2	54.3	-32.5
	E5-800-2	132.03	117.18	53.28			E5D-800-2	62.6	53.4	4.3
	E5-800-3	131.33	116.78	55.48			E5D-800-3	62.4	52.6	5.0
R	E5-010-1	0.21	0.26	0.05		R	E5D-010-1	-0.1	-0.3	-0.3
R dup	E5-010-1	0.23	0.18	0.05		R	E5D-010-2	0.0	0.0	BDL
R	E5-010-2	0.16	0.16	0.04		R	E5D-010-3	0.1	0.0	BDL
R	E5-010-3	0.21	0.18	0.07						
R	E5-100-1	2.23	2.01	0.61		R	E5D-100-1	1.3	1.0	-0.2
R	E5-100-2	2.10	1.81	0.63		R	E5D-100-2	1.2	0.9	-0.1
R	E5-100-3	2.19	1.81	0.43		R	E5D-100-3	1.4	0.8	-0.3
R	E5-400-1	72.58	67.04	30.34		R	E5D-400-1	38.0	37.7	-0.8
R	E5-400-2	71.58	66.29	30.34		R	E5D-400-2	38.1	35.2	6.7
R	E5-400-3	74.73	67.74	29.39		R	E5D-400-3	40.9	36.2	5.3
R	E5-800-1	131.23	117.53	53.63		R	E5D-800-1	52.9	54.4	-31.1
R	E5-800-2	133.08	115.78	54.03		R	E5D-800-2	63.4	52.2	4.2
R	E5-800-3	133.43	116.73	57.08		R	E5D-800-3	65.0	52.9	5.9

Test 3 - Surface Soil Heterogeneity						Test 3 - Surface Soil Heterogeneity				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	**Sample**	**2,4-DNT**	**2,6-DNT**	**NG**		**Other**	**Sample**	**2,4-DNT**	**2,6-DNT**	**NG**
Echo range #1	E6-E1-1	13.80	12.65	5.75			E6D-E1-1	8.5	7.8	3.5
R	E6-E1-1	13.10	11.60	5.25		R	E6D-E1-1	7.8	6.8	3.0
	E6-E1-2	12.60	11.55	5.40			E6D-E1-2	7.3	5.8	3.2
R	E6-E1-2	12.55	10.25	5.60		R	E6D-E1-2	7.1	4.5	3.3
	E6-E1-3	12.85	11.20	5.10			Rep 3 Soils Sacrificed for Soil Analysis			
dup	**E6-E1-3**	12.60	11.40	5.55			Rep 3 Soils Sacrificed for Soil Analysis			
R	**E6-E1-3**	12.90	10.95	5.65			Rep 3 Soils Sacrificed for Soil Analysis			
R dup	**E6-E1-3**	12.55	10.10	6.25			Rep 3 Soils Sacrificed for Soil Analysis			
Echo range #2	E6-E2-1	12.25	10.90	4.65			E6D-E2-1	7.3	6.5	2.2
R	E6-E2-1	12.25	10.20	4.55		R	E6D-E2-1	7.3	5.8	2.1
	E6-E2-2	12.00	10.90	5.00			E6D-E2-2	7.1	5.5	3.3
R	E6-E2-2	11.60	10.25	4.60		R	E6D-E2-2	6.7	4.9	3.0
	E6-E2-3	12.85	10.70	5.20			Rep 3 Soils Sacrificed for Soil Analysis			
R	E6-E2-3	13.40	10.65	6.15			Rep 3 Soils Sacrificed for Soil Analysis			
Juliet range #1	E6-J1-1	13.70	11.80	5.05			E6D-J1-1	8.3	6.5	2.0
R	E6-J1-1	13.35	12.25	4.45		R	E6D-J1-1 (E6-J1-2 C	7.9	6.8	1.3
	E6-J1-2	14.30	12.25	5.25			E6D-J1-2	8.3	5.9	2.4
dup	**E6-J1-2**	14.60	12.20	6.75		R	E6D-J1-2	8.5	5.9	3.9
R	**E6-J1-2**	14.15	12.60	5.45						
R dup	**E6-J1-2**	14.45	11.75	5.75						
	E6-J1-3	14.65	13.00	5.65			Rep 3 Soils Sacrificed for Soil Analysis			
R	E6-J1-3	14.90	12.30	5.10			Rep 3 Soils Sacrificed for Soil Analysis			
Juliet range #2	E6-J2-1	18.35	15.85	7.95			E6D-J2-1	12.7	9.7	3.4
R	E6-J2-1	18.75	15.95	7.80		R	E6D-J2-1	13.0	9.8	3.2
	E6-J2-2	18.95	16.80	7.30			E6D-J2-2	11.5	9.7	2.7
R	E6-J2-2	18.95	16.45	7.60		R	E6D-J2-2	11.5	9.3	2.9
	E6-J2-3	19.45	17.15	7.80			Rep 3 Soils Sacrificed for Soil Analysis			
R	E6-J2-3	19.70	16.45	8.25			Rep 3 Soils Sacrificed for Soil Analysis			
Kilo range,west	E6-K6-1	16.05	14.35	6.85			E6D-K6-1	9.6	7.9	2.3
R	E6-K6-1	16.35	13.80	6.40		R	E6D-K6-1	9.8	7.4	1.8
	E6-K6-2	16.65	15.05	7.65			E6D-K6-2	9.2	7.4	2.9
R	E6-K6-2	16.95	13.80	6.65		R	E6D-K6-2	9.3	6.1	1.9
	E6-K6-3	17.25	15.50	6.80			Rep 3 Soils Sacrificed for Soil Analysis			
R	E6-K6-3	17.40	14.65	6.70			Rep 3 Soils Sacrificed for Soil Analysis			

Test 4 - Depth Evaluation						Test 4 - Depth Evaluation				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
Kilo range 9-12"	E7-K3-1	9.90	7.08	3.88			E7D-K3-1	6.6	0.0	-3.0
R	E7-K3-1	9.85	8.08	3.83		R	E7D-K3-1	6.4	0.9	-3.0
	E7-K3-2	9.65	6.38	2.53			E7D-K3-2	6.0	-0.7	-3.5
R	E7-K3-2	9.85	6.53	3.03		R	E7D-K3-2	6.2	-0.5	-3.0
	E7-K3-3	9.50	7.13	2.88			E7D-K3-3	6.1	-0.1	-3.8
R	E7-K3-3	8.80	6.78	3.18		R	E7D-K3-3	5.4	-0.3	-3.4
Kilo range 18-24"	E7-K4-1	7.25	4.68	2.23			E7D-K4-1	3.5	-4.0	-8.0
dup	E7-K4-1	7.20	4.38	2.38		R	E7D-K4-1	3.4	-4.4	-7.8
R	E7-K4-1	6.80	3.98	1.48			E7D-K4-2	5.1	-2.4	-5.7
R dup	E7-K4-1	6.70	4.23	2.18		R	E7D-K4-2	5.0	-2.4	-6.1
	E7-K4-2	6.70	3.78	1.83			E7D-K4-3	5.8	-2.5	-6.0
R	E7-K4-2	6.75	3.83	1.28		R	E7D-K4-3	5.7	-2.7	-5.3
	E7-K4-3	7.65	4.23	1.83						
R	E7-K4-3	7.50	3.93	2.53						
Kilo range 30-36"	E7-K5-1	5.55	3.33	2.23			E7D-K5-1	4.9	-2.1	-5.6
R	E7-K5-1	5.70	3.63	2.18		R	E7D-K5-1	5.0	-1.9	-5.7
	E7-K5-2	6.00	2.83	2.38			E7D-K5-2	5.3	-2.7	-5.5
R	E7-K5-2	5.50	3.33	1.98		R	E7D-K5-2	4.8	-2.3	-5.7
	E7-K5-3	5.30	2.48	1.93			E7D-K5-3	4.8	-2.9	-6.0
R	E7-K5-3	5.00	2.03	1.63		R	E7D-K5-3	4.5	-3.3	-6.2
Echo range 9-12"	E7-E3-1	10.80	11.73	3.18			E7D-E3-1	7.4	9.5	3.2
R	E7-E3-1	10.90	11.98	2.88		R	E7D-E3-1	7.5	9.8	3.0
	E7-E3-2	10.30	10.98	2.43			E7D-E3-2	7.1	8.9	2.6
R	E7-E3-2	10.20	10.68	4.73		R	E7D-E3-2	7.0	8.6	4.6
	E7-E3-3	10.50	11.43	3.13			E7D-E3-3	7.3	9.2	3.2
R	E7-E3-3	10.25	11.03	3.23		R	E7D-E3-3	7.0	8.9	3.3
Echo range 18-24"	E7-E4-1	6.55	9.23	4.03			E7D-E4-1	4.8	8.4	4.5
R	E7-E4-1	6.50	8.88	3.73		R	E7D-E4-1	4.8	8.1	4.2
	E7-E4-2	5.80	9.03	-0.32			E7D-E4-2	4.3	8.2	0.6
R	E7-E4-2	6.35	8.73	2.03		R	E7D-E4-2	4.8	8.0	2.8
	E7-E4-3	5.65	8.93	1.88			E7D-E4-3	4.1	8.1	2.8
R	E7-E4-3	5.85	7.93	3.28		R	E7D-E4-3	4.3	7.2	4.0
Echo range 30-36	E7-E5-1	6.90	8.78	1.28			E7D-E5-1	5.1	7.8	1.8
R	E7-E5-1	7.80	8.58	3.28		R	E7D-E5-1	6.0	7.7	3.8
	E7-E5-2	8.25	9.98	2.93			E7D-E5-2	6.4	9.1	3.4
R	E7-E5-2	8.45	9.48	3.43		R	E7D-E5-2	6.7	8.5	3.9
	E7-E5-3	8.40	9.93	1.73			E7D-E5-3	6.2	8.6	2.0
R	E7-E5-3	9.35	10.83	2.43		R	E7D-E5-3	7.1	9.5	2.7
Juliet range 9-12"	E7-J3-1	7.10	9.68	1.58			E7D-J3-1	4.1	8.2	1.1
R	E7-J3-1	7.55	7.78	3.98		R	E7D-J3-1	4.6	6.5	3.5
	E7-J3-2	7.90	9.88	2.03			E7D-J3-2	5.0	8.5	2.1
R	E7-J3-2	7.25	9.13	3.03		R	E7D-J3-2	4.3	7.7	3.0
	E7-J3-3	8.35	10.08	2.53			E7D-J3-3	5.0	8.5	2.1
R	E7-J3-3	8.70	9.88	3.13		R	E7D-J3-3	5.4	8.3	2.8
Juliet range 18-24"	E7-J4-1	6.65	6.48	1.73			E7D-J4-1	4.3	6.6	1.7
R	E7-J4-1	6.25	6.93	0.32		R	E7D-J4-1	3.9	7.1	0.4
	E7-J4-2	6.20	7.03	1.33			E7D-J4-2	3.9	7.2	1.3
R	E7-J4-2	6.40	6.88	1.33		R	E7D-J4-2	4.1	7.0	1.3
	E7-J4-3	6.50	6.88	0.38			E7D-J4-3	4.1	6.9	0.5
R	E7-J4-3	6.45	6.93	1.73		R	E7D-J4-3	4.1	7.0	1.8
Juliet range 30-36"	E7-J5-1	2.85	6.53	1.88			E7D-J5-1	3.0	7.6	2.7
R	E7-J5-1	2.70	5.63	1.83		R	E7D-J5-1	3.0	6.9	2.6
	E7-J5-2 repeat	4.15	6.53	1.38			E7D-J5-2 repeat	2.3	7.2	0.1
R	E7-J5-2 repeat	2.60	6.23	0.27		R	E7D-J5-2 repeat	3.0	7.3	0.3
R dup	E7-J5-2 repeat	3.25	6.28	0.53			E7D-J5-3 repeat	2.9	3.7	1.5
	E7-J5-3 repeat	2.80	2.28	0.88		R	E7D-J5-3 repeat	3.8	8.0	2.6
R	E7-J5-3 repeat	3.70	6.88	2.08						

Test 5 - Temperature Evaluation						Test 5 - Temperature Evaluation				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
12ºC	E8-T12-1	20.09	15.94	6.81			E8D-T12-1	12.9	10.1	-1.6
dup	E8-T12-1	20.24	16.19	7.06			E8D-T12-2	14.3	11.4	-1.1
	E8-T12-2	22.49	18.04	7.36			E8D-T12-3	13.9	10.9	-1.8
	E8-T12-3	22.09	17.49	7.71						
32ºC	E8-T32-1	17.84	13.84	6.81			E8D-T32-1	8.0	6.0	-3.3
	E8-T32-2	17.49	13.14	6.66			E8D-T32-2	9.4	7.0	-1.6
	E8-T32-3	18.44	13.64	7.56			E8D-T32-3	9.2	6.1	-1.6
R	E8-T12-1	19.79	15.39	6.81		R	E8D-T12-1	12.5	9.4	-2.0
dup	E8-T12-1	20.09	15.39	6.66		R	E8D-T12-2	14.1	10.7	-1.4
R	E8-T12-2	22.29	17.44	7.31		R	E8D-T12-3	13.9	10.7	-2.8
R	E8-T12-3	22.09	17.44	7.01						
R	E8-T32-1	17.84	13.04	6.76		R	E8D-T32-1	8.0	5.2	-3.5
R	E8-T32-2	17.64	13.24	7.66		R	E8D-T32-2	9.5	6.9	-1.0
R	E8-T32-3	18.34	13.49	6.61		R	E8D-T32-3	9.1	5.9	-2.9

Test 6 - pH						Test 6 - pH				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
pH=4.2	E9-pH4-1	23.33	25.48	16.58			E9D-PH4-1	15.4	13.8	1.6
Dup	E9-pH4-1	23.48	25.63	17.58		R	E9D-PH4-1	15.5	13.9	2.7
R	E9-pH4-1	23.38	25.38	17.33			E9D-PH4-2	15.2	13.7	1.6
R dup	E9-pH4-1	23.68	25.98	17.98		R	E9D-PH4-2	15.4	13.7	2.1
	E9-pH4-2	23.53	25.58	17.48			E9D-PH4-3	15.6	13.7	2.9
R	E9-pH4-2	23.58	25.58	17.63		R	E9D-PH4-3	16.0	14.4	3.2
	E9-pH4-3	23.38	25.53	17.28						
R	E9-pH4-3	23.78	26.18	17.23						
pH=8.2	E9-PH9-1	25.80	26.14	14.77			E9D-pH9-1	17.0	10.6	2.2
R	E9-PH9-1	26.16	27.48	15.62		R	E9D-pH9-1	17.4	11.9	3.1
	E9-PH9-2	24.55	25.54	13.93			E9D-pH9-2	15.9	10.3	1.8
R	E9-PH9-2	25.07	25.76	14.78		R	E9D-pH9-2	16.5	10.5	2.6
	E9-PH9-3	25.04	27.05	14.56			E9D-pH9-3	16.0	11.5	2.0
R	E9-PH9-3	25.44	27.13	15.68		R	E9D-pH9-3	16.3	11.5	3.2

Test 7 - Desorption of Unfired Propellant						Test 7 - Desorption of Unfired Propellant				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
	Not Applicable						Not Applicable			

Test 8 - Desorption of Contaminated Soil						Test 8 - Desorption of Contaminated Soil				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
	Not Applicable						Not Applicable			

Test 9 - Repeat of Rainwater Test						Test 9 - Repeat of Rainwater Test				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
w/o biocide	NB-rain-1	31.70	19.93	17.70			NB-R1D	32.6	25.2	BDL
	NB-rain-2	28.05	18.18	13.50			NB-R2D	BDL	BDL	15.8
	NB-rain-3	28.25	18.83	15.00			NB-R3D	28.9	23.0	BDL
With biocide	YB-rain-1	20.70	16.08	7.15			YB-R1D	14.8	16.8	4.3
	YB-rain-2	21.75	17.48	6.80			YB-R2D	16.3	18.7	4.7
	YB-rain-3	21.65	17.53	7.80			YB-R3D	15.9	18.4	5.5

Test 10 - Fired Propellant						Test 10 - Fired Propellant				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
	Not Applicable						Not Applicable			

Test 11 - Biocide Comparison						Test 11 - Biocide Comparison				
	Soil Concentration S_o (mg/kg) at end of Sorption Test						Corrected Soil Concentration (mg/kg) at end of Desorption Test			
Other	Sample	2,4-DNT	2,6-DNT	NG		Other	Sample	2,4-DNT	2,6-DNT	NG
Glutaraldehyde	T13-R1	10.33	12.25	4.95			T13D-R1	6.3	9.8	5.2
	T13-R2	7.93	10.50	5.00			T13D-R2	4.7	8.6	5.5
	T13-R3	9.08	11.25	4.95			T13D-R3	5.8	9.3	5.4
HgCl	T14-R1	11.88	13.55	5.00			T14D-R1	10.0	11.1	5.9
	T14-R2	9.88	12.65	4.55			T14D-R2	7.8	10.2	5.8
	T14-R3	11.13	14.25	5.25			T14D-R3	9.0	11.3	6.0

Abbreviations
Sample ID-C = Control = Initial spike concentration (Co)
Sample ID-B = Blank = DI
R = Reverse order (some sets of samples were analyzed in both forward and reverse order on the HPLC)
dup = analytical duplicate
BDL = below MDL

Appendix I

Column Results

Column 1A - Aqueous NG/DNT with Biocide																	
					Influent	Effluent	Influent				Effluent						
Vial Number	Test	Tracer Elapsed Time (hrs)	NG/DNT Elasped Time (hrs)	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	1,2-GDN (mg/L)	1,3-GDN (mg/L)	Comments
0	T	0			0	0									
1	T	0.5			100										Begin Cl (100 ppm) Tracer Sorption Test.
6	T	3				57.8									
10	T	5				66.7									
14	T	7				75.1									
17	T	8.5				81.6									
22	T	11				85.6									
26	T	13				87.3									
30	T	15				89.1									
34	T	17				90.7									
38	T	19				91.3									
42	T	21				92.0									
46	T	23				93.2									
50	T	25			100	99.2									
54	T	27				98.2									
58	T	29				100.5									
62	T	31				98.1									
66	T	33				99.6									
70	T	35				99.2									
74	T	37				99.1									
82	T	41				97.1									
94	T	47				105.6									
108	T	54			100	102.7									
122	T	61				102.0									
136	T	68				100.1									
146	T	73				99.1									
156	T	78			100	96.4									
170	T	85				95.2									
194	T	97			100	98.5									
244	T	122			100	94.5									
285	T	142.5				88.3									
295	T	147.5			100										End of Cl sorption tracer test
1	NG	148	0.42	0.04	0		0.971	1.022	0.985	BDL	BDL	BDL	BDL	BDL	Begin NG/DNT (1 ppm) Sorption Test, Begin Cl Desorption Tracer Test.
3	NG	148.84	1.26	0.12		87.8									
5	NG	149.68	2.1	0.21						BDL	BDL	BDL	BDL	BDL	
7	NG	150.52	2.94	0.30		88.2									
11	NG	152.2	4.62	0.47		83.9									
12	NG	152.62	5.04	0.52						BDL	BDL	BDL	BDL	BDL	
15	NG	153.88	6.3	0.65		73.6									
16	NG	154.3	6.72	0.69						BDL	BDL	BDL	BDL	BDL	
19	NG	155.56	7.98	0.82		62.3									
23	NG	157.24	9.66	1.00		52.7									
24	NG	157.66	10.08	1.04						0.186 J	BDL	BDL	BDL	BDL	
27	NG	158.92	11.34	1.17		45.4									
28	NG	159.34	11.76	1.22						0.185 J	BDL	BDL	BDL	BDL	
29	NG	159.76	12.18	1.26						0.186 J	BDL	BDL	BDL	BDL	
31	NG	160.6	13.02	1.35		39.3									
35	NG	162.28	14.7	1.52		37.1									
36	NG	162.7	15.12	1.57						0.311	BDL	BDL	BDL	BDL	
39	NG	163.96	16.38	1.70		29.9									
42	NG	165.22	17.64	1.83						0.404	BDL	BDL	BDL	BDL	
44	NG	166.06	18.48	1.92		25.2									
48	NG	167.74	20.16	2.15	0		0.959	0.958	0.984	0.532	0.025 J	BDL	BDL	BDL	
52	NG	169.42	21.84	2.27		20.7									
56	NG	171.1	23.52	2.44			0.966	1.020	0.967						
58	NG	171.94	24.36	2.53						0.532	BDL	BDL	BDL	BDL	
60	NG	172.78	25.2	2.62		19.1									

					Column 1A - Aqueous NG/DNT with Biocide										
					Influent	Effluent	Influent			Effluent					
Vial Number	Test	Tracer Elapsed Time (hrs)	NG/DNT Elasped Time (hrs)	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	1,2-GDN (mg/L)	1,3-GDN (mg/L)	Comments
71	NG	177.4	29.82	3.10		15.7									
72	NG	177.82	30.24	3.14						0.595	0.100 J	0.166 J	BDL	BDL	
84	NG	182.86	35.28	3.67		12.2									
87	NG	184.12	36.54	3.80						0.595	0.100 J	0.166 J	BDL	BDL	
96	NG	187.9	40.32	4.19						0.622	0.163	0.212	BDL	BDL	
98	NG	188.74	41.16	4.28	0	9.1									
116	NG	196.3	48.72	5.06						0.622	0.163	0.212	BDL	BDL	
119	NG	197.56	49.98	5.20		6.7									
120	NG	197.98	50.4	5.24						0.642	0.198	0.216	BDL	BDL	
143	NG	207.64	60.06	6.24		5.4									
144	NG	208.06	60.48	6.29						0.653	0.209	0.239	BDL	BDL	
145	NG	208.48	60.9	6.33	0					0.642	0.198	0.216	BDL	BDL	
168	NG	218.14	70.56	7.34						0.667	0.221	0.231	BDL	BDL	
173	NG	220.24	72.66	7.56		4.0				0.653	0.209	0.239	BDL	BDL	End of Cl Desorption Test
203	NG		85.26	8.87						0.667	0.231	0.221	BDL	BDL	
225	NG		94.5	9.83						0.721	0.250	0.250	BDL	BDL	
	NG		95.34	9.92			0.973	0.964	0.972						
226	NG		119.7	12.45						0.761	0.286	0.293	BDL	BDL	
227	NG		145.74	15.16						0.807	0.334	0.338	BDL	BDL	
	NG		146.16	15.21			0.966	0.963	0.973						
228	NG		170.94	17.78						0.864	0.399	0.383	BDL	BDL	
229	NG		192.78	20.06			0.983	1.025	0.979	0.842	0.426	0.426	BDL	BDL	
	NG		194.88	20.28			0.994	0.994	0.956						
230	NG		268.38	27.93			0.961	1.019	0.950	0.874	0.424	0.419	BDL	BDL	
	NG		268.8	27.97			0.960	0.966	0.943						
231	NG		483.84	50.35			0.999	0.940	0.883						
232	NG		484.26	50.40						0.906	0.499	0.467	BDL	0.104 J	End of NG/DNT Sorption Tests, Stopped NG/DNT Influent.
233	NG		485.52	50.53			BDL	BDL	BDL	0.915	0.492	0.447	0.062 J	BDL	Begin NG/DNT Desorption Test. Started DI + Biocide Influent.
237	NG		487.2	50.70						0.909	0.487	0.467	0.060 J	BDL	
245	NG		490.56	51.05						0.897	0.498	0.481	BDL	BDL	
253	NG		493.92	51.40						0.866	0.491	0.451	BDL	BDL	
261	NG		497.28	51.75						0.779	0.493	0.468	BDL	BDL	
269	NG		500.64	52.10						0.661	0.495	0.467	0.059 J	BDL	
277	NG		504	52.45						0.558	0.497	0.459	BDL	BDL	
289	NG		509.04	52.98						0.450	0.486	0.467	0.062 J	BDL	
297	NG		512.4	53.33						0.405	0.462	0.447	BDL	BDL	
305	NG		515.76	53.67						0.393	0.475	0.437	BDL	BDL	
309	NG		517.44	53.85						0.378	0.441	0.413	BDL	BDL	
316	NG		527.94	54.94						0.384	0.456	0.417	BDL	BDL	
325	NG		531.72	55.34						0.373	0.436	0.401	BDL	BDL	
	NG		628.97	63.84			BDL	0.034 J	0.044 J						
326	NG		628.97	63.84						0.167 J	0.314	0.288	BDL	BDL	
	NG		628.97	63.84			0.078 J	BDL	BDL						
	NG		701.22	70.16			BDL	0.070 J	0.069 J						
327	NG		767.47	75.95						0.059 J	0.223	0.222	BDL	BDL	
	NG		843.22	82.57			BDL	0.029 J	BDL						
	NG		869.72	84.89			BDL	0.022 J	BDL						
	NG		1007.387	96.92			BDL	BDL	BDL						
	NG		1079.22	103.20			BDL	BDL	BDL						
	NG		1105.97	105.54			BDL	BDL	BDL						
330	NG		1108.22	105.74						BDL	0.061 J	0.045 J	BDL	BDL	
	NG		1108.52	105.77			BDL	0.022 J	BDL						
	NG		1204.22	114.13			BDL	BDL	BDL						
331	NG		1204.22	114.13						BDL	0.046 J	BDL	BDL	BDL	
332	NG		1269.72	119.86						BDL	0.042 J	BDL	BDL	BDL	
	NG		1269.72	119.86			BDL	BDL	BDL						End of Column Test

Column 1B - Aqueous NG/DNT with Biocide																	
					Influent	Effluent	Influent			Effluent							
Vial Number	Test	Tracer Elapsed Time (hrs)	NG/DNT Elasped Time (hrs)	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	1,2-GDN (mg/L)	1,3-GDN (mg/L)	Comments
T0	T	0.0			0	0									
T1	T	0.4			50										Start of Cl (50 ppm) Sorption Tests
T5	T	2.1				50.7									
T18	T	7.6				44.4									
T28	T	11.8				44.7									
38	T	16.0				45.7									
49	T	20.6				45.9									
60	T	25.2			50	46.0									
71	T	29.8				45.8									
86	T	36.1				45.3									
101	T	42.4			50	45.0									
T113	T	47.5				45.4									
1	NG	48.7	0.42	0.04	0		0.973	1.002	0.967	BDL	BDL	BDL	BDL	BDL	Start of NG/DNT (1ppm) Sorption Test. Begin Cl Desorption Test.
3	NG	49.6	1.26	0.12		47.1									
8	NG	51.7	3.36	0.34		44.3									
10	NG	52.5	4.20	0.43						BDL	BDL	BDL	BDL	BDL	
12	NG	53.3	5.04	0.52		33.0									
17	NG	55.4	7.14	0.74		21.2									
19	NG	56.3	7.98	0.82						BDL	BDL	BDL	BDL	BDL	
22	NG	57.5	9.24	0.95		14.5									
27	NG	59.6	11.34	1.17		10.4									
29	NG	60.5	12.18	1.26						0.189 J	BDL	BDL	BDL	BDL	
32	NG	61.7	13.44	1.39		7.9									
36	NG	63.4	15.12	1.57		6.6									
38	NG	64.3	15.96	1.65						0.354	BDL	BDL	BDL	BDL	
41	NG	65.5	17.22	1.79		5.2									
46	NG	67.6	19.32	2.00		4.3									
48	NG	68.5	20.16	2.09	0					0.448	BDL	BDL	BDL	BDL	
51	NG	69.7	21.42	2.22		3.5									
53	NG	70.6	22.26	2.31			0.983	1.025	0.979						
55	NG	71.4	23.10	2.40		3.1									
58	NG	72.7	24.36	2.53						0.543	0.021 J	0.050 J	BDL	BDL	
62	NG	74.3	26.04	2.70		2.5									
67	NG	76.4	28.14	2.92						0.581	BDL	0.073 J	BDL	BDL	
72	NG	78.5	30.24	3.14		1.7									
77	NG	80.6	32.34	3.36						0.580	0.074 J	0.127 J	BDL	BDL	
81	NG	82.3	34.02	3.53		1.3									
87	NG	84.8	36.54	3.80						0.569	0.100 J	0.149 J	BDL	BDL	
90	NG	86.1	37.80	3.93		1.0									
96	NG	88.6	40.32	4.19						0.589	0.137	0.184 J	BDL	BDL	
99	NG	89.9	41.58	4.32	0	0.8									
106	NG	92.8	44.52	4.63						0.598	0.162	0.193 J	BDL	BDL	
109	NG	94.1	45.78	4.76		0.7									
111	NG	94.9	46.62	4.85			0.95	0.92	0.93						
115	NG	96.6	48.30	5.02						0.617	0.164	0.197 J	BDL	BDL	
116	NG	97.0	48.72	5.06			0.994	0.994	0.956						
118	NG	97.9	49.56	5.15		0.6									
133	NG	104.2	55.86	5.81		0.5									
144	NG	108.8	60.48	6.29						0.748	0.203	0.235	BDL	BDL	
147	NG	110.0	61.74	6.42		0.4									
172	NG	120.5	72.24	7.51	0	0.3									End of Cl Desorption Test
173	NG	121.0	72.66	7.56						0.754	0.233	0.244	BDL	BDL	
202	NG		84.84	8.82						0.797	0.244	0.264	BDL	BDL	
230	NG		96.60	10.05						0.819	0.268	0.267	BDL	BDL	
257	NG		107.94	11.23						0.808	0.294	0.301	BDL	BDL	
281	NG		118.02	12.28			0.961	1.019	0.950						
282	NG		118.44	12.31			0.960	0.966	0.943	0.777	0.317	0.320	BDL	BDL	
283	NG		146.35	14.75						0.697	0.271	0.275	BDL	BDL	
289	NG		336.35	31.37			0.999	0.940	0.883						
	NG						0.945	0.904	1.060						
290	NG		384.52	35.58			0.951	0.905	1.007	0.777	0.355	0.362	BDL	BDL	
291	NG		482.19	44.12			0.894	0.842	0.970	0.795	0.393	0.379	BDL	BDL	
292	NG		529.85	48.28			BDL	0.070 J	0.069 J	0.820	0.413	0.402	BDL	BDL	Began NG/DNT Desorption Test, Switched influent to DI water + biocide
296	NG		531.85	48.46						0.855	0.424	0.421	BDL	BDL	
300	NG		533.85	48.63						0.801	0.404	0.401	BDL	BDL	
304	NG		535.85	48.81						0.793	0.416	0.382	BDL	BDL	
308	NG		537.85	48.98						0.802	0.411	0.394	BDL	BDL	

Column 1B - Aqueous NG/DNT with Biocide																
					Influent	Effluent	Influent			Effluent						
Vial Number	Test	Tracer Elapsed Time (hrs)	NG/DNT Elasped Time (hrs)	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	1,2-GDN (mg/L)	1,3-GDN (mg/L)	Comments
316	NG		541.85	49.33						0.776	0.418	0.387	BDL	BDL	
324	NG		545.85	49.68						0.730	0.413	0.402	BDL	BDL	
332	NG		549.85	50.03						0.616	0.422	0.396	BDL	BDL	
344	NG		555.85	50.56						0.532	0.419	0.411	BDL	BDL	
356	NG		561.85	51.08						0.436	0.410	0.384	BDL	BDL	
368	NG		567.85	51.61						0.375	0.400	0.381	BDL	BDL	
380	NG		573.85	52.13						0.377	0.392	0.368	BDL	BDL	
404	NG		585.85	53.18						0.306	0.373	0.335	BDL	BDL	
428	NG		597.85	54.23						0.293	0.353	0.312	BDL	BDL	
452	NG		609.85	55.28						0.292	0.333	0.301	BDL	BDL	
484	NG		625.85	56.68						0.246 J	0.320	0.281	BDL	BDL	
	NG		648.02	58.61			BDL	BDL	BDL						
	NG		696.35	62.84			BDL	0.029 J	BDL						
	NG		722.85	65.16			BDL	0.022 J	BDL						
	NG		860.52	77.19			BDL	BDL	BDL						
	NG		932.35	83.47			BDL	BDL	BDL						
	NG		959.10	85.81			BDL	BDL	BDL						
487	NG		961.35	86.01						BDL	0.162	0.166 J	BDL	BDL	
	NG		961.69	86.04			BDL	0.101	0.090 J						
	NG		1057.35	94.40			BDL	0.085 J	0.093 J						
488	NG		1057.35	94.40						0.066 J	0.117	0.121 J	BDL	BDL	
489	NG		1122.85	100.13						0.077 J	0.128	0.110 J	BDL	BDL	
	NG		1122.85	100.13			BDL	0.073 J	0.042 J						End of Column Test

Column 2A - Aqueous NG/DNT without Biocide																
					Influent	Effluent	Influent			Effluent						
Vial Number	Test	Tracer Elapsed Time (hrs)	NG/DNT Elasped Time (hrs)	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	1,2-GDN (mg/L)	1,3-GDN (mg/L)	Comments
0	T	0			0	0									
1	T	0.42			50										Start Cl (50 ppm) sorption test.
2	T	0.84				19.6									
8	T	3.36				19.3									
12	T	5.04				18.6									
16	T	6.72				25.0									
20	T	8.4				34.4									
24	T	10.08				42.1									
28	T	11.76				46.1									
32	T	13.44				47.8									
36	T	15.12				46.7									
42	T	17.64				47.2									
48	T	20.16			50	47.4									
60	T	25.2				46.7									
72	T	30.24				47.5									
96	T	40.32				48.7									
107	T	44.94			50	49.8									End Cl Sorption Test.
108	T	45.36													
1	NG	45.86	0.5	0.04	0					BDL	BDL	BDL	BDL	BDL	Start NG/DNT (1 ppm) Sorption Test, Start Cl Desorption Test.
4	NG	47.36	2	0.17		50.9									
11	NG	50.86	5.5	0.48		49.4				BDL	BDL	BDL	BDL	BDL	
19	NG	54.86	9.5	0.83		29.0									
23	NG	56.86	11.5	1.01						BDL	BDL	BDL	BDL	BDL	
27	NG	58.86	13.5	1.18		14.9									
35	NG	62.86	17.5	1.53		9.2				BDL	BDL	BDL	BDL	BDL	
44	NG	67.36	22	1.92		6.1									
47	NG	68.86	23.5	2.05						BDL	BDL	BDL	BDL	BDL	
53	NG	71.86	26.5	2.32	0	4.1									
59	NG	74.86	29.5	2.58						BDL	BDL	BDL	BDL	BDL	
63	NG	76.86	31.5	2.75		2.6									
71	NG	80.86	35.5	3.10		1.8				BDL	BDL	BDL	BDL	BDL	
81	NG	85.86	40.5	3.54		1.1									
83	NG	86.86	41.5	3.63						BDL	BDL	BDL	BDL	BDL	
91	NG	90.86	45.5	3.98		0.6									
95	NG	92.86	47.5	4.15						BDL	BDL	BDL	BDL	BDL	
106	NG	98.36	53	4.63	0	0.3									
108	NG	99.36	54	4.72		49.8									
116	NG	103.36	58	5.07		0.2									End of Cl desorption test.
	NG		79	6.91						BDL	BDL	BDL	BDL	BDL	
148	NG		121.5	10.62			1.090	0.999	0.958	BDL	BDL	BDL	BDL	BDL	
	NG		144.5	12.63			0.969	0.915	1.080						
149	NG		169.5	14.82			0.999	0.961	1.042						
150	NG		267	23.34			1.064	1.029	1.159	BDL	BDL	BDL	BDL	BDL	
151	NG		405.5	35.45			1.000	0.972	0.882						
	NG		433.1667	37.87			1.140	1.134	1.052						
152	NG		506.5	44.28			0.982	1.000	0.914						
154	NG		746.5	65.27			1.020	1.005	0.937	BDL	BDL	BDL	BDL	BDL	
	NG		790	69.07			0.999	1.035	0.908						
	NG		842.5	73.66			0.753	0.389	0.369						
155	NG		842.5	73.66						BDL	BDL	BDL	BDL	BDL	
156	NG		908	79.39						BDL	BDL	BDL	BDL	BDL	
	NG		908	79.39			0.701	0.348	0.349						
	NG		912	79.74			0.968	0.949	0.864						
157	NG		1004	87.78						0.147 J	0.022 J	BDL	0.050 J	BDL	
	NG		1004.5	87.82			0.615	0.255	0.247						
158	NG		1106.5	96.74						BDL	BDL	BDL	BDL	BDL	
159	NG		1272.333	111.24						BDL	BDL	BDL	BDL	BDL	
	NG		1273	111.30			10.43	10.4	10.11						Increased NG/DNT Influent to 10 ppm
160	NG		1295.583	113.27						BDL	0.043 J	0.229	BDL	BDL	
161	NG		1342.5	117.37						BDL	BDL	0.086 J	BDL	BDL	
	NG		1344.083	117.51			9.611	9.271	8.418						
162	NG		1437.783	125.70						BDL	BDL	BDL	BDL	BDL	
163	NG		1655.5	144.74			4.766	2.017	2.063	BDL	BDL	BDL	BDL	BDL	
164	NG		1756	153.53						BDL	BDL	BDL	BDL	BDL	Increased NG/DNT Influent to 100 ppm
165	NG		1756.5	153.57						BDL	BDL	BDL	BDL	BDL	
166	NG		1757	153.61						BDL	BDL	BDL	BDL	BDL	
167	NG		1757.5	153.66						BDL	BDL	BDL	BDL	BDL	
168	NG		1765	154.31						0.057 J	BDL	BDL	0.505	0.249 J	
	NG		1827	157.00											End of Column Test

Column 2B - Aqueous NG/DNT without Biocide																	
					Influent	Effluent	Influent			Effluent							
Vial Number	Test	Tracer Elapsed Time (hrs)	NG/DNT Elasped Time (hrs)	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	1,2-GDN (mg/L)	1,3-GDN (mg/L)	Comments
0	T	0			0	0.0									
1	T	0.42			50										Start of Cl (50 ppm) Sorption Test
4	T	1.68				14.5									
8	T	3.36				19.8									
12	T	5.04				13.5									
16	T	6.72				18.1									
20	T	8.4				28.3									
24	T	10.08				34.8									
28	T	11.76				37.6									
32	T	13.44				38.8									
36	T	15.12				39.6									
42	T	17.64				40.7									
48	T	20.16			50	42.8									
60	T	25.2				45.6									
72	T	30.24				47.6									
96	T	40.32			50	48.4									
108	T	45.36				48.7									End of Sorption Test
1	NG	45.86	0.5	0.04	0					BDL	BDL	BDL	BDL	BDL	Start NG/DNT (1 ppm) Sorption Test, Start Cl Desorption Tests
5	NG	47.86	2.5	0.22		52.0									
11	NG	50.86	5.5	0.48		51.4									
12	NG	51.36	6	0.52						BDL	BDL	BDL	BDL	BDL	
18	NG	54.36	9	0.79		33.6									
24	NG	57.36	12	1.05						BDL	BDL	BDL	BDL	BDL	
27	NG	58.86	13.5	1.18		15.5									
35	NG	62.86	17.5	1.53		8.6									
36	NG	63.36	18	1.57						BDL	BDL	BDL	BDL	BDL	
42	NG	66.36	21	1.84		5.3									
48	NG	69.36	24	2.10						BDL	BDL	BDL	BDL	BDL	
51	NG	70.86	25.5	2.23	0	2.5									
60	NG	75.36	30	2.62						BDL	BDL	BDL	BDL	BDL	
62	NG	76.36	31	2.71		0.8									
72	NG	81.36	36	3.15						BDL	BDL	BDL	BDL	BDL	
75	NG	82.86	37.5	3.28		0.3									
84	NG	87.36	42	3.67						BDL	BDL	BDL	BDL	BDL	
87	NG	88.86	43.5	3.80		0.2									
96	NG	93.36	48	4.20						BDL	BDL	BDL	BDL	BDL	
99	NG	94.86	49.5	4.33	0	0.2									
120	NG	105.36	60	5.25						BDL	BDL	BDL	BDL	BDL	
123	NG	106.86	61.5	5.38		0.2									
144	NG	117.36	72	6.29						BDL	BDL	BDL	BDL	BDL	
147	NG	118.86	73.5	6.43	0	0.2									End Cl Desorption Test
148	NG		121.50	10.62			1.090	0.999	0.958	BDL	BDL	BDL	BDL	BDL	
	NG		144.50	12.63			0.969	0.915	1.080						
149	NG		169.50	14.82			1.010	0.944	1.032	BDL	BDL	BDL	BDL	BDL	
150	NG		267.00	23.34			1.064	1.029	1.159	BDL	BDL	BDL	BDL	BDL	
	NG		315.08	27.55			0.976	0.902	0.860						
151	NG		405.50	35.45						BDL	BDL	BDL	BDL	BDL	
	NG		433.17	37.87			1.140	1.134	1.052						
	NG		481.50	42.10			1.000	0.972	0.882						
	NG		508.00	44.41			0.982	1.000	0.914						
	NG		596.33	52.14			1.020	1.005	0.937						
	NG		744.25	65.07			0.999	1.035	0.908						
154	NG		746.50	65.27						BDL	BDL	BDL	BDL	BDL	
	NG		746.90	65.30			0.942	0.951	0.862						
	NG		842.50	73.66			0.977	0.948	0.894						
155	NG		842.50	73.66						BDL	BDL	BDL	BDL	BDL	
156	NG		908.00	79.39						BDL	BDL	BDL	BDL	BDL	
	NG		908.00	79.39			0.952	0.880	0.850						
	NG		912.00	79.74			0.968	0.949	0.864						
157	NG		1004.00	87.78						0.094 J	BDL	BDL	BDL	BDL	End Column Test

Column 3A - Propellent Residue with Biocide																
				Influent	Effluent	Influent			Effluent							
Vial Number	Tracer Elapsed Time (hrs)	NG/DNT Elasped Time (hrs)	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)		2,4-DNT (mg/L)		2,6-DNT (mg/L)	1,2-GDN (mg/L)		1,3-GDN (mg/L)		Comments
	0	0	0	0	0	0	0	0										
1	0.5	0.5	0.04	50		BDL	BDL	BDL										Start of Cl Sorption Test and Propellant Dissolution/Sorption/Desorption
4	2	2	0.17						BDL		BDL		BDL	BDL		BDL		
6	3	3	0.26		1.3													
8	4	4	0.35		1.2													
9	4.5	4.5	0.39						BDL		BDL		BDL	BDL		BDL		
16	8	8	0.70		12.9													
17	8.5	8.5	0.74						BDL		BDL		BDL	0.632		BDL		
24	12	12	1.05		30.0													
25	12.5	12.5	1.09						BDL		BDL		BDL	0.683		BDL		
32	16	16	1.40		39.2													
33	16.5	16.5	1.44						BDL		BDL		BDL	BDL		BDL		
35	17.5	17.5	1.53			BDL	BDL	BDL										
40	20	20	1.75		28.2													
41	20.5	20.5	1.79						BDL		BDL		BDL	BDL		BDL		
48	24	24	2.10		45.5													
	24.5	24.5	2.14			BDL	BDL	BDL										
49	24.5	24.5	2.14	50					BDL		BDL		BDL	BDL		BDL		
56	28	28	2.45		47.4													
57	28.5	28.5	2.49						BDL		BDL		BDL	0.542		BDL		
64	32	32	2.80		47.4													
65	32.5	32.5	2.84						BDL		BDL		BDL	0.109	J	BDL		
72	36	36	3.15		47.3													
73	36.5	36.5	3.19						BDL		BDL		BDL	BDL		BDL		
80	40	40	3.50		47.5													
81	40.5	40.5	3.54						BDL		BDL		BDL	BDL		BDL		
88	44	44	3.85		46.9													
89	44.5	44.5	3.89						BDL		BDL		BDL	BDL		BDL		
96	48	48	4.20		47.1													
97	48.5	48.5	4.24						BDL		BDL		BDL	BDL		BDL		
104	52	52	4.55	50	47.1													
105	52.5	52.5	4.59						BDL		BDL		BDL	BDL		BDL		
112	56	56	4.90		46.6													
113	56.5	56.5	4.94						BDL		BDL		BDL	BDL		BDL		
120	60	60	5.25		46.6													
121	60.5	60.5	5.29						BDL		BDL		BDL	0.154	J	BDL		
128	64	64	5.60		45.8													
129	64.5	64.5	5.64						BDL		BDL		BDL	0.138	J	BDL		
136	68	68	5.95		46.6													
137	68.5	68.5	5.99						BDL		BDL		BDL	0.14	J	BDL		
160	80	80	6.99		46.0													
161	80.5	80.5	7.04						BDL		0.021	J	BDL	0.126	J	BDL		
183	93.75	93.75	8.20			BDL	BDL	BDL										
184	94.25	94.25	8.24	50	47.1													
185	94.75	94.75	8.28	0					0.21	J	BDL		BDL	0.132	J	BDL		End of Cl Sorption Test
191	97.75	97.75	8.55						BDL		BDL		BDL	BDL		BDL		
	139.25	139.25	12.17			BDL	BDL	BDL										
	165.75	165.75	14.49			BDL	BDL	BDL										
192	168	168	14.69						0.102	J	BDL		BDL	BDL		BDL		
	168.417	168.417	14.72			BDL	BDL	BDL										
	264	264	23.08			BDL	BDL	BDL										
193	264	264	23.08	0					0.135	J	BDL		BDL	BDL		BDL		
194	329.5	329.5	28.81						0.067	J	BDL		BDL	BDL		BDL		
194	329.5	329.5	28.81						0.108	J	BDL		BDL	0.052	J	BDL		
	329.5	329.5	28.81															
	333.5	333.5	29.16			BDL	BDL	BDL										
195	425.5	425.5	37.20						BDL		BDL		BDL	BDL		BDL		
	426	426	37.24			BDL	BDL	BDL										
196	528	528	46.16						0.111	J	BDL		BDL	BDL		BDL		
197	693.833	693.833	60.66						0.088	J	BDL		BDL	BDL		BDL		
	694.5	694.5	60.72			BDL	BDL	BDL										
	765.583	765.583	66.93			BDL	BDL	BDL										
198	909	909	79.47						0.140	J	BDL		BDL	BDL		BDL		
199	1077	1077	94.16			0.250	BDL	BDL	0.085	J	BDL		BDL	BDL		BDL		
	1249	1249	109.00															End of Column Test

Column 3B - Propellent Residue with Biocide															
				Influent	Effluent	Influent				Effluent					
Vial Number	Tracer Elapsed Time (hrs)	NG/DNT Elasped Time (hrs)	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	1,2-GDN (mg/L)	1,3-GDN (mg/L)	Comments
0	0	0	0.00	0	0									
1	0.5	0.5	0.04			BDL	BDL	BDL						Started Cl (50 ppm) Sorption Test, and Propellant Dissolution/Sorption/Desorption Test
2	1	1	0.09						BDL	BDL	BDL	BDL	BDL	
6	3	3	0.26		2.0961									
9	4.5	4.5	0.39						BDL	BDL	BDL	BDL	BDL	
14	7	7	0.61		21.146									
17	8.5	8.5	0.74						BDL	BDL	BDL	BDL	BDL	
19	9.5	9.5	0.83		30.838									
25	12.5	12.5	1.09						BDL	BDL	BDL	BDL	BDL	
27	13.5	13.5	1.18		37.328									
33	16.5	16.5	1.44						BDL	BDL	BDL	BDL	BDL	
35	17.5	17.5	1.53		40.553	BDL	BDL	BDL						
41	20.5	20.5	1.79						BDL	BDL	BDL	BDL	BDL	
46	23	23	2.01		44.415									
47	23.5	23.5	2.05						0.630	BDL	BDL	BDL	BDL	
48	24	24	2.10			0.093 J	BDL	BDL						
51	25.5	25.5	2.23		45.871									
57	28.5	28.5	2.49						BDL	BDL	BDL	BDL	BDL	
59	29.5	29.5	2.58		47.875									
65	32.5	32.5	2.84						BDL	BDL	BDL	BDL	BDL	
67	33.5	33.5	2.93		48.241									
73	36.5	36.5	3.19						BDL	BDL	BDL	BDL	BDL	
81	40.5	40.5	3.54						BDL	BDL	BDL	BDL	BDL	
83	41.5	41.5	3.63		48.476									
89	44.5	44.5	3.89						BDL	BDL	BDL	BDL	BDL	
91	45.5	45.5	3.98		48.301									
97	48.5	48.5	4.24						BDL	BDL	BDL	BDL	BDL	
99	49.5	49.5	4.33		48.779									
105	52.5	52.5	4.59						BDL	BDL	BDL	BDL	BDL	
107	53.5	53.5	4.68		48.54									
113	56.5	56.5	4.94						BDL	BDL	BDL	BDL	BDL	
115	57.5	57.5	5.03		48.398									
118	59	59	5.16		48.398									
121	66.5	66.5	5.81						BDL	BDL	BDL	BDL	BDL	
123	67.5	67.5	5.90		46.803									
129	70.5	70.5	6.16						BDL	BDL	BDL	BDL	BDL	
131	71.5	71.5	6.25		47.71									
137	74.5	74.5	6.51						BDL	BDL	BDL	BDL	BDL	
139	75.5	75.5	6.60		47.069									
152	93.75	93.75	8.20			BDL	BDL	BDL						
159	97.25	97.25	8.50		48.967									End Cl Sorption Test
160	97.75	97.75	8.55						BDL	BDL	BDL	BDL	BDL	
	139	139	12.15			BDL	BDL	BDL						
	165.75	165.75	14.49			BDL	BDL	BDL						
161	168	168	14.69						BDL	BDL	BDL	BDL	BDL	
	168.5	168.5	14.73			BDL	BDL	BDL						
	264	264	23.08			BDL	BDL	BDL						
162	329.5	329.5	28.81						BDL	BDL	BDL	0.066 J	BDL	
	329.5	329.5	28.81			BDL	BDL	BDL						
	333.5	333.5	29.16			BDL	BDL	BDL						
163	425.5	425.5	37.20						BDL	BDL	BDL	0.059 J	BDL	
	426	426	37.24			BDL	BDL	BDL						
164	528	528	46.16						0.102 J	BDL	BDL	0.051 J	BDL	
	693.833	693.833	60.66						0.085 J	BDL	BDL	BDL	BDL	
	694.5	694.5	60.72			BDL	BDL	BDL						
	765.583	765.583	66.93			BDL	BDL	BDL						
	909	909	79.47						0.100 J	BDL	BDL	BDL	BDL	
	1077	1077	94.16			0.110 J	BDL	BDL	0.078 J	BDL	BDL	BDL	BDL	
	1080.5	1080.5	94.47											End Column Test

Column 4A - Propellent Residue without Biocide													
			Influent	Effluent	Influent			Effluent					
Vial Number	and NG/DNT Elasped	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	1,2-GDN (mg/L)	1,3-GDN (mg/L)	Comments
0	0	0.00	0	0	0	0	0						Start Cl (50 ppm) Sorption Test, Propellant Dissolution/Sorption/Desorption Test
	0	0.00			BDL	BDL	BDL						
1	0	0.00	46.5										
4	2	0.17						BDL	BDL	BDL	BDL	BDL	
8	4	0.35						BDL	BDL	BDL	0.083 J	BDL	
12	6	0.52						BDL	BDL	BDL	0.138 J	BDL	
16	8	0.70						BDL	BDL	BDL	0.191 J	0.159 J	
20	10	0.87						BDL	BDL	BDL	0.216 J	0.180 J	
24	12	1.05						BDL	BDL	BDL	0.257	0.207 J	
27	13.5	1.18		51.8									
28	14	1.22						BDL	BDL	BDL	0.244 J	0.183 J	
32	16	1.40						BDL	BDL	BDL	0.147 J	0.109 J	
36	18	1.57						BDL	BDL	BDL	BDL	BDL	
40	20	1.75						BDL	BDL	BDL	BDL	BDL	
44	22	1.92						BDL	BDL	BDL	BDL	BDL	
48	24	2.10						BDL	BDL	BDL	BDL	BDL	
52	26	2.27	50.0					BDL	BDL	BDL	BDL	BDL	
56	28	2.45						BDL	BDL	BDL	BDL	BDL	
83	41.5	3.63						BDL	BDL	BDL	BDL	BDL	
85	42.5	3.72						BDL	BDL	BDL	BDL	BDL	
89	44.5	3.89						BDL	BDL	BDL	BDL	BDL	
92	46	4.02	50.0	48.0									
93	46.5	4.07						BDL	BDL	BDL	BDL	BDL	
132	66	5.77		47.2									
133	66.5	5.81						BDL	BDL	BDL	BDL	BDL	
136	68	5.95		48.9									
138	69	6.03						BDL	BDL	BDL	BDL	BDL	
140	70	6.12		46.9									
145	72.5	6.34		47.2									
146	73	6.38						BDL	BDL	BDL	BDL	BDL	
149	74.5	6.51	50.0	47.3									
153	76.5	6.69		46.9									
154	77	6.73						BDL	BDL	BDL	BDL	BDL	
157	78.5	6.86		47.4									
161	80.5	7.04		46.6									
162	81	7.08						BDL	BDL	BDL	BDL	BDL	
165	82.5	7.21		47.3									
170	85	7.43						BDL	BDL	BDL	BDL	BDL	
176	88	7.69						BDL	BDL	BDL	BDL	BDL	
	89.5	7.82			BDL	BDL	BDL						
180	90	7.87	46.9										
183	91.5	8.00		48.1									
184	92	8.04						BDL	BDL	BDL	BDL	BDL	
187	93.5	8.17		49.6									
191	95.5	8.35		49.3									End Cl Sorption Test
192	96	8.39	0.0					BDL	BDL	BDL	BDL	BDL	Start Cl Desorption Test
195	97.5	8.52		33.5									
199	99.5	8.70		18.4									
200	100	8.74	0.0					BDL	BDL	BDL	BDL	BDL	
203	101.5	8.87		9.6									
208	104	9.09						BDL	BDL	BDL	BDL	BDL	
212	106	9.27						BDL	BDL	BDL	BDL	BDL	
220	110	9.62						BDL	BDL	BDL	BDL	BDL	
223	111.5	9.75		3.7									
228	114	9.97		3.4									
229	114.5	10.01						BDL	BDL	BDL	BDL	BDL	
232	116	10.14		3.0									
236	118	10.32		2.3									
237	118.5	10.36						BDL	BDL	BDL	BDL	BDL	
240	120	10.49		1.6									
244	122	10.67	0.0	1.2									End Cl Desorption Test
245	122.5	10.71						BDL	BDL	BDL	BDL	BDL	
250	191.75	16.76						BDL	BDL	BDL	BDL	BDL	
251	357.583	31.26						BDL	BDL	BDL	BDL	BDL	
	358.25	31.32			BDL	BDL	BDL						
	429.333	37.54			BDL	BDL	BDL						
252	740.75	64.76			BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	

Column 4B - Propellent Residue without Biocide														
			Influent	Effluent	Influent			Effluent						
Vial Number	Tracer and NG/DNT Elasped Time (hrs)	NG/DNT Pore Volume	Cl (mg/L)	Cl (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	NG (mg/L)	2,4-DNT (mg/L)	2,6-DNT (mg/L)	1,2-GDN (mg/L)	1,3-GDN (mg/L)	Comments
0	0	0.00	0	0.0	0	0	0						Start CL (50 ppm) Sorption Test, Propellant Dissolution/Sorption/Desorption Test
1	0.25	0.04	50					BDL	BDL	BDL	BDL	BDL	
17	4.25	0.74		29.8									
25	6.25	1.09		35.5									
29	7.25	1.27		36.6									
33	8.25	1.44		37.1									
37	9.25	1.62		40.0									
41	10.25	1.79		41.1									
48	12.00	2.10						BDL	BDL	BDL	BDL	BDL	
50	12.50	2.19	50										
65	16.25	2.84		42.1									
83	20.75	3.63		45.2									
91	22.75	3.98		46.1									
96	24.00	4.20						BDL	BDL	BDL	BDL	BDL	
99	24.75	4.33		47.0									
100	25.00	4.37	50										
102	25.50	4.46		48.3									End Cl Sorption Test
106	26.50	4.63		35.6									Start Cl Desorption Test
118	29.50	5.16		15.8									
122	30.50	5.33		13.0									
134	33.50	5.86		8.7									
138	34.50	6.03		7.5									
142	35.50	6.21		6.7									
144	36.00	6.29						BDL	BDL	BDL	BDL	BDL	
147	36.75	6.43	0	5.7									
151	37.75	6.60		4.8									
167	41.75	7.30		2.6									
175	43.75	7.65		1.6									
184	46.00	8.04			BDL	BDL	BDL						
191	47.75	8.35		0.5									
192	48.00	8.39						BDL	BDL	BDL	BDL	BDL	
199	49.75	8.70	0	0.4									
200	189.25	33.09						BDL	BDL	BDL	BDL	BDL	
201	357.25	62.47			BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	
	625	109											End Column Tests

Appendix J

Column soil results.

			Soil Concentration (mg/kg)				
Sample ID	**Column**	**Depth (cm)**	**NG**	**2,4-DNT**	**2,6-DNT**	**1,2-GDN**	**1,3-GDN**
1A-1 soil	1A	0-2	2.094	0.228	0.106	0.156	BDL
1A-2 soil	1A	2-4	BDL	0.204	0.052	BDL	BDL
1A-3 soil	1A	4-6	BDL	0.256	0.114	0.156	BDL
1A-4 soil	1A	6-8	BDL	0.364	0.068	0.122	BDL
1A-5 soil	1A	8-10	0.058 J	0.334	0.168	0.196	BDL
1A-6 soil	1A	10-12	BDL	0.366	0.188	0.140	BDL
1A-7 soil	1A	12-14	BDL	0.386	0.194	0.108	BDL
1A-8 soil	1A	14-16	0.098	0.394	0.180	0.230	BDL
1A-9 soil	1A	16-18	0.090	0.470	0.212	BDL	BDL
1A-10 soil	1A	18-20	BDL	0.456	0.090	BDL	BDL
1B-0-2cm	1B	0-2	BDL	0.708	0.382	BDL	BDL
1B-2-4cm	1B	2-4	0.062 J	0.554	0.302	BDL	BDL
1B-4-6cm	1B	4-6	0.042 J	0.640	0.358	BDL	BDL
1B-6-8cm	1B	6-8	0.06 J	0.652	0.358	BDL	BDL
1B-8-10cm	1B	8-10	0.108	0.642	0.364	0.122	BDL
1B-10-12cm	1B	10-12	BDL	0.614	0.366	0.094	BDL
1B-12-14cm	1B	12-14	0.168	0.690	0.440	BDL	BDL
2B-1-Soil	2B	0-2	BDL	BDL	0.060	BDL	BDL
2B-2-Soil	2B	2-4	0.336	0.556	0.534	BDL	BDL
2B-3-Soil	2B	4-6	0.160	BDL	BDL	BDL	BDL
2B-4-Soil	2B	6-8	BDL	BDL	BDL	BDL	BDL
2B-5-Soil	2B	8-10	0.106	BDL	BDL	BDL	BDL
2B-6-Soil	2B	10-12	0.152	BDL	BDL	BDL	BDL
2B-7-Soil	2B	12-14	0.04 J	BDL	BDL	BDL	BDL
2B-8-Soil	2B	14-16	BDL	BDL	BDL	BDL	BDL
2B-9-Soil	2B	16-18	BDL	BDL	BDL	BDL	BDL
2B-10-Soil	2B	18-20	BDL	BDL	BDL	BDL	BDL
3B-0-2cm	3B	0-2	518	BDL	BDL	BDL	BDL
3B-2-4cm	3B	2-4	100	BDL	BDL	BDL	BDL
3B-4-6cm	3B	4-6	1.76	BDL	BDL	0.066	BDL
3B-6-8cm	3B	6-8	0.640	BDL	BDL	0.086	BDL
3B-8-10cm	3B	8-10	0.472	BDL	BDL	0.070	BDL
3B-10-12cm	3B	10-12	0.500	BDL	BDL	0.080	BDL
3B-12-14cm	3B	12-14	0.256	BDL	BDL	0.044 J	BDL
3B-14-16cm	3B	14-16	0.224	BDL	BDL	0.062 J	BDL
3B-16-18cm	3B	16-18	0.420	0.286	0.272	0.100	BDL
3B-18-20cm	3B	18-20	0.510	BDL	BDL	0.078	BDL
BDL = below detection limit, J = estimated value							

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14. ABSTRACT

The mobility potential of nitroglycerin (NG) and dinitrotoluene (DNT) in small-arms range (SAR) soils was studied through a determination of adsorption and desorption. Measured laboratory batch-adsorption soil/water partitioning coefficients (Kds) for 2,4-DNT, 2,6-DNT, and NG ranged from 0.1 to 21.3, 0 to 18.2, and 0 to 7.3 L/kg, respectively. Mean adsorption Kd for 2,4-DNT and 2,6-DNT were 3.2 and 2.6 L/kg, respectively. The mean value for NG was 0.9 L/kg. The variables impacting adsorption were organic matter and cation exchange. Unfired and fired propellant tests suggest that NG and DNT is not completely available for dissolution, and tests with weathered soils indicate none of the NG is available, although analysis shows NG is still present in the soil. Dissolution is the most important process in describing migration of deposited propellant compounds from SARs. Once released from nitrocellulose and dissolved in water, adsorption and degradation processes further limit NG and DNT mobility. Column experiments were conducted to augment batch tests. Nearly instantaneous breakthrough of NG was evident for the pair of columns containing aqueous NG/DNT with biocide. Results for the columns containing aqueous NG/DNT without biocide, fresh-fired propellant residue with biocide, and fresh-fired propellant residue without biocide indicated no breakthrough.

15. SUBJECT TERMS

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